STIC-ILL

From:

Flood, Michele

Sent: Wednesday, October 16, 2002 9:49 AM

STIC-ILL

To: Subject:

Following articles needed. Mailbox 11D13. Thanks.

416487

09/428,203

Journal of Pharmaceutical Research and Development (1998), 3(2), 75-79. Ekpendu, T. O. E etal. Chemical constituents of the extractives of Napoleona imperialis P. Beauv. (Lecythidaceae).

ACTA CRYSTALLOGR SECT B STRUCT CRYSTALLOGR CRYST CHEM, (1980) 36 (7), 1593-1598.). SPIRLET M R etal. STRUCTURE OF ACETYLATED NAPOLEOGENIN.

Phytochemistry (1980), 19(4), 615-22. Kapundu, Mpuza etal. New triterpenoids from Napoleonaea imperialis.

67) 634 AZL 10/17-RC

Acta

Crystallographica

Pada Memedaneil Madaellas airl Padile has ind Cycnines

ઇ=U732∀(198**0**)

TSSN 0567#74

Les auteurs remercient Mlle José Piquion du Laboratoire de Chimie Organique de la Faculté des Sciences d'Abidjan qui a extrait ce composé; la Direction de l'Office Central de Mécanographie d'Abidjan (Côte d'Ivoire) qui nous a permis d'effectuer les calculs sur ses ordinateurs IBM 370-145. L'un de mous (JPD) remercie le Fonds National de la Recherche Scientifique pour le mandat dont il a bénéficié.

Références

BAGGIO, R., WOOLFSON, M. M., DECLERCQ, J. P. & GERMAIN, G. (1978). Acta Cryst. A 34, 883-892.

BARTON, D. H. R. & KIRBY, G. W. (1960). Proc. Chem. Soc. pp. 392-393.

Barton, D. H. R. & Kirby, G. W. (1962). J. Chem. Soc. pp. 806-817.

bon, H. G. & DÖPKE, W. (1960). Naturwissenschaften, 47,

Not, H. G., DÖPKE, W. & BEITNER, A. (1957). Chem. Ber. 90, 2197-2202.

BOIT, H. G. & EHMKE, H.₁(1956). Chem. Ber. **89**, 163–167.

CRUICKSHANK, D. W. J. (1965). Computing Methods in Crystallography, edite par J. S. ROLLETT, pp. 113-114. Oxford: Pergamon Press.

FALES, H. M., GIUFFRIDA, L. D. & WILDMAN, W. C. (1956). J. Am. Chem. Soc. 78, 4145-4150.

JOHNSON, C. K. (1965). ORTEP. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.

KLYNE, W. & PRELOG, V. (1960). Experientia, 16, 521-523.
LAIHO, S. M. & FALES, H. M. (1964). J. Am. Chem. Soc. 86, 4434-4438.

Roques, R. & Lapasset, J. (1976). Acta Cryst. B32, 579-582.

Roques, R., Lapasset, J., Rogers, D. & Williams, D. J. (1976). *Acta Cryst.* B32, 3358.

STEWART, J. M., KRUGER, G. J., AMMON, H. L., DICKINSON,
C. & HALL, S. R. (1972). The XRAY system – version
of June 1972. Tech. Rep. TR-192. Computer Science
Center, Univ. of Maryland, College Park, Maryland.

WILLIAMS, D. J. & ROGERS, D. (1964). Proc. Chem. Soc. p. 357.

Acta Cryst. (1980). B36, 1593-1598

ď

五百八百五百日

The Structure of Acetylated Napoleogenin

By M. R. Spirlet

Laboratoires de Cristallographie et de Physique Expérimentale, Université de Liège au Sart Tilman, B 4000 Liège, Belgium

L. DUPONT AND O. DIDEBERG

Institut de Physique B5, Université de Liège au Sart Tilman, B 4000 Liège, Belgium

AND M. KAPUNDU

Laboratoire de Chimie Organique, Institut de Chimie B6, Université de Liège au Sart Tilman, B 4000 Liège, Belgium

(Received 18 October 1979; accepted 11 February 1980)

Abstract

Actylated napoleogenin, $C_{58}H_{84}O_{18}$, crystallizes in space group $P2_1$ with two molecules in a unit cell of emensions $a=19\cdot127$ (1), $b=10\cdot812$ (1), $c=14\cdot879$ (1) Å, $\beta=102\cdot540$ (2)°. The crystal structure as solved by direct methods. Full-matrix least-squares refinement with all atoms treated isotropically and sing 3139 reflections gave an R value of $0\cdot108$. The compound possesses a triterpene skeleton with ciscusted D and E rings in the chair form. A 3,4-10-angelyl-6-deoxy- β -glucopyranosyl group sits at C(21) on the E ring. The molecular structure of

0567-7408/80/071593-06\$01.00

acetylated napoleogenin is formulated by the present analysis as 21β -(2-O-acetyl-3,4-di-O-angelyl-6-deoxy- β -glucopyranosyloxy)-3 β ,16 α ,22 α ,24,28-pentaacetoxy-olean-12-ene. The large distortion of the whole molecular skeleton may result from 1,3 diaxial interaction. The packing is mainly dictated by close van der Waals contacts between the molecules.

Introduction

Within the framework of chemical taxonomy studies, the acetylated form of napoleogenin, a new pentacyclic © 1980 International Union of Crystallography

triterpene, was isolated from the seeds of Napoleonaea imperialis, a plant native to Zaire. An X-ray crystal-structure investigation of the compound was undertaken in order to establish unequivocally its chemical structure and stereochemistry.

Table 1. Atomic positional and isotropic thermal parameters with standard deviations in acetylated napoleogenin

	x	y	z	$B(\dot{A}^2)$
(i) Triter	pene nucleus			
C(1)	0.5868 (7)	0.047(1)	-0.0589 (9)	6-3 (3)
C(2)	0.5595 (8)	0.063 (2)	-0·164 (1)	7.2 (3)
C(3)	0.6094 (7)	-0.002 (2)	-0.214(1)	6·1 (3)
C(4)	0.6195 (7)	-0·142 (2)	-0·1966 (9)	5.9 (3)
C(5)	0.6442 (6)	-0.159(1)	-0·1917 (8)	5.1 (2)
C(6)	0.6609 (6)	-0.290(1)	-0.0571 (8)	4.8 (2)
C(7)	0.7072 (7)	-0.287(1)	0.0403 (9)	5-5 (3)
C(8)	0.6706 (6)	–0·222 (1)	0.1111 (7)	4.4 (2)
C(9)	0.6418 (6)	-0.094 (1)	0.0712 (7)	4.4 (2)
C(10)	0.5957 (6)	-0.096 (1)	-0.0314 (8)	4.8 (2)
C(11)	0.6039 (7)	-0.027 (2)	0.136(1)	6.8 (3)
C(12)	0.6383 (7)	-0.045 (2)	0.2365 (9)	5.7 (3)
C(13)	0.6943 (6)	-0.124(1)	0.2691 (8)	4.8 (2)
C(14)	0.7283 (6)	-0·196 (1)	0.2040 (7)	4.3 (2)
C(15)	0.7571 (6)	-0.319(1)	0.2482 (8)	5.1 (2)
C(16)	0-7920 (6)	-0·319 (1)	0.3532 (8)	4.7 (2)
C(17)	0.7468 (6)	-0.253 (1)	0.4107 (7)	4.4 (2)
C(18)	0.7208 (6)	-0.124(1)	0.3740 (8)	4.4 (2)
C(19)	0.7756 (5)	-0·022 (1)	0.4064 (7)	4.4 (2)
C(20)	0.8003 (6)	-0·010 (1)	0.5115 (8)	4.6 (2)
C(21)	0.8372 (5)	-0·137 (1)	0.5414 (7)	3.8 (2)
C(22)	0.7860 (6)	-0·242 (1)	0.5156 (7)	4.5 (2)
(ii) Extra	anuclear			
C(23)	0.6816 (9)	-0.188 (2)	-0·241 (1)	8.0 (4)
C(24)	0.5513 (7)	-0.207 (1)	0·244 (1)	6.2 (3)
C(25)	0.5199 (8)	-0·150 (2)	-0.042 (1)	6.8 (3)
C(26)	0.6107 (7)	-0.301(2)	0.1299 (9)	5.8 (3)
C(27)	0.7924 (6)	-0.117(1)	0.1880 (8)	5.2 (2)
C(28)	0.6778 (6)	-0.329(1)	0.4023 (8)	5.1 (2)
C(29)	0.8561 (7)	0.095 (2)	0.531 (1)	6.0 (3)
C(30)	0.7390 (7)	0.020(2)	0.5577 (9)	5.7 (3)
C(31)	0.5960 (9)	0.132 (2)	0.348 (1)	7.7 (4)
C(32)	0.553 (1)	(,	0.445 (2)	10.5 (6)
C(33)	0.523 (1)	-0·422 (2)	-0·291 (1)	8.5 (4)
C(34)	0.549 (1)	0·545 (2)	-0·281 (2)	10.6 (5)
C(35)	0.9223 (7)	-0.314 (2)	0·3678 (9) 0·379 (1)	5·9 (3) 9·0 (4)
C(36)	0.983 (1)	-0·231 (2) -0·545 (2)	0.379(1)	8·4 (4)
C(37)	0.6493 (9)	-0.545 (2) -0.665 (2)	0.439 (2)	10.7 (5)
C(38)	0.673 (1)	-0.430 (2)	0.439 (2)	6.9 (3)
C(39) C(40)	0·8109 (8) 0·866 (1)	-0.436(2) -0.536(2)	0.617(1)	9.5 (5)
. ,	0.5805 (6)	0.018 (1)	-0·3126 (7)	7.7 (2)
O(1) O(2)	0.6359 (7)	0.200 (2)	-0·3110 (9)	10.2 (3)
O(3)	0.5631 (6)	-0.340(1)	-0·2356 (7)	7.5 (2)
O(3)	0.4668 (8)	-0.378(2)	-0·343 (1)	11.5 (4)
O(4) O(5)	0.8612 (4)	-0.378(2) -0.252(1)	0.3665 (5)	4.3 (1)
O(6)	0.9218 (5)	-0.426(1)	0.3673 (7)	7.7 (2)
O(7)	0.6974 (5)	-0·458 (1)	0.4258 (6)	5-8 (2)
O(8)	0.597(1)	-0.517(2)	0.332(1)	15.3 (5)
O(9)	0.8288 (4)	-0.353(1)	0.5383 (5)	5.0(2)
O(10)	0.7681 (7)	-0.412(1)	0.6413 (8)	9.0 (3)
O(11)	0.8596 (4)	-0.138(1)	0.6416 (5)	4.5 (1)
/			\- /	

	^	,	٠,	D (n)
(iii) Gluc	opyranosyl mo	oiety	•	
Č(1')	0.9320 (6)	-0·128 (1)	0-6775 (7)	4.3 (2
C(2')	0.9452 (6)	-0.079(1)	0.7747 (8)	4.5 (1
C(3')	1.0269 (5)	-0.072 (1)	0.8153 (8)	4-7(2
C(4')	1.0603 (6)	-0·192 (1)	0.8042 (8)	4·7(4 Our
C(5')	1.0391 (7)	-0.240(1)	0.7026 (9)	5.6(legenin
O(1')	0.9637 (4)	-0.247 (1)	0.6774 (5)	4 1 1
C(6')	1.0692 (8)	-0.370 (2)	0.691 (1)	6-40 B -am
C(7')	1.1814 (8)	-0·212 (2)	0.897(1)	6.4(1 gluco
C(8')	1.260(1)	-0.187 (2)	0.893 (1)	8·2 More
C(9')	1.301 (1)	-0·156 (3)	0.977 (2)	11-2 group
C(10')	1.2873 (7)	-0.142 (2)	1.0605 (9)	9·1(at C
C(11')	1.279 (1)	-0 ⋅192 (3)	0.798 (2)	11.2(
C(7")	1.0759 (8)	0.047 (2)	0.948 (1)	6.5 (Figlocat
C(8")	1.078 (1)	0.061 (2)	1.053 (1)	8-8 meth
C(9")	1.0416 (7)	0.015 (2)	1.0960 (9)	8.0 dedu
C(10")	0.9722 (7)	-0.058 (2)	1.0686 (9)	9.1 lated
C(11")	1 · 142 (1)	0-149 (2)	1-093 (1)	8.8()
C(12')	0.8652 (7)	0.064 (2)	0.817 (1)	0.0
C(13')	0.840(1)	0-196 (2)	0.807(1)	8.8 (olean
O(2')	1-1383 (4)	–0⋅172 (1)	0.8199 (5)	5.60 in F
O(3')	1-1558 (8)	–0⋅258 (2)	0.956(1)	11.0(11.0) in Fi
O(2")	1.0318 (4)	-0.051 (1)	0.9124 (6)	5.6 (4 PLU
O(3")	1-1033 (8)	0.116 (2)	0.907(1)	AU-UDMAC.
O(4')	0.9187 (4)	0.044 (1)	0.7743 (5)	5.4 lengt
O(5')	0.8376 (6)	-0.014 (1)	0-8567 (9)	8.9(1 • Li

Table 1 (cont.)

Experimental

deposi tary P throug

lograp

Colorless needle-shaped crystals, elongated along the axis, were grown from methanol.

The space group was unequivocally established by Weissenberg and precession photographs.

Cell parameters and intensities were measured with four-circle Hilger diffractometer using Cu $K\alpha$ radiatio ($\lambda = 1.5418$ Å, $\omega - 2\theta$ scan). Integrated intensities were recorded for 3353 independent reflections. They were corrected for Lorentz and polarization effects but not for absorption (μ for Cu $K\alpha = 0.72$ mm⁻¹).

The structure was solved by direct methods using MULTAN (Main, Lessinger, Woolfson, Germain) Declercq, 1977). A large number of cycles of structure factor and Fourier calculations were necessary to fin all the atoms. The oxygen atoms were identified with the help of chemical and structural consideration Refinement of atomic coordinates and individu isotropic thermal parameters for C and O atoms carried out by a full-matrix least-squares method (program SFLS, Prewitt, 1967) on 3139 reflections which $I > 2\sigma(I)$. In the last cycles of refinement, atoms were included in calculated positions assumi C-H bond lengths of 1.087 A. All were given same isotropic temperature factor $B = 4.0 \text{ A}^{23}$ Cruickshank (1961) type of weighting scheme used. The final value of the conventional discrepand factor R is 0.108. We could not carry out further refinement with anisotropic thermal parameters: large number of variables would have involved

ch computing time and expense. Our final positional method thermal parameters are listed in Table 1.*

Discussion

(7), (8), (8), (8), (9), (5),

2) (9)

21199111516159

4-3 (2)

4·5 (2) 4·7 (2)

10-0 (3)

5-4 (2) 8-9 (3)

ted along the b

established by

ieasured with a ii Kα radiation intensities were ins. They were effects but not

methods using ų, Germain & les of structure cessary to find identified with considerations. and individual 1. O atoms was juares meth^{od} 9 reflections for refinement, H itions assuming were given the $= 4.0 \text{ Å}^2. \text{ }^{\text{A}}$ ોંgંscheme w^છી nal discrepancy rry out furths parameters: the e involved too

Official results indicate that acetylated napoleomin is a pentacyclic oleanene-type triterpene of the imyrin series with a 3,4-di-o-angelyl-6-deoxy-βpucopyranosyl group located on the E ring at C(21). Moreover, in the triterpene nucleus, a geminal dimethyl group is situated at C(20) and four other methyl groups at C(4), C(8), C(10) and C(14). Acetyl groups are located at C(3), C(16) and C(22) while two acetoxymethyl groups are found at C(4) and C(17). We deduced that acetylated napoleogenin should be formulated as 21β -(2-O-acetyl-3,4-di-O-angelyl-6-deoxy- β glucopyranosyloxy) - 3β , 16α , 22α , 24, 28 - pentaacetoxyolean-12-ene. The numbering of the atoms is illustrated Fig. 1. The molecular conformation is shown in Fig. 2, a stereodrawing produced by the program PLUTO (Crystallographic Data Files, 1979). Bond lengths and angles are listed in Tables 2 and 3.

Lists of structure factors and hydrogen coordinates have been exposited with the British Library Lending Division as Supplementry Publication No. SUP 35161 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystalbraphy, 5 Abbey Square, Chester CH1 2HU, England.

Fig. 1. Numbering of atoms in acetylated napoleogenin.

Table 2. Bond lengths (A) with standard deviations in parentheses

(i) Triterpene nuc	lauc	(ii) Extranuclear	
C(1)—C(2)	1·54 (2)	C(3)-O(1)	1.47 (2)
	1.60 (2)	C(4)—C(23)	1.56 (2)
C(1)–C(10)		C(4)—C(24)	1.51 (2)
C(2)—C(3)	1.51 (2)	C(4)-C(24) C(8)-C(26)	1.51 (2)
C(3)—C(4)	1.54 (2)		1.54 (2)
C(4)-C(5)	1.54 (2)	C(10)-C(25)	1.56 (2)
C(5)-C(6)	1.52 (2)	C(14)—C(27)	
C(5)-C(10)	1.58 (2)	C(16)-O(5)	1.48 (1)
C(6)-C(7)	1.53 (2)	C(17)-C(28)	1.54 (2)
C(7)-C(8)	1.56 (2)	C(20)-C(29)	1.54 (2)
C(8)-C(9)	1-56 (2)	C(20)-C(30)	1.52 (2)
C(8)-C(14)	1.59 (2)	C(21)-O(11)	1.46 (1)
C(9)-C(10)	1.59 (2)	C(22)-O(9)	1.45 (2)
C(9)-C(11)	1-51 (2)	C(31)–O(1)	1.40 (2)
C(11)-C(12)	1-51 (2)	C(31)=O(2)	1.11 (2)
C(12)=C(13)	1.37 (2)	C(31)-C(32)	1.50 (3)
C(13)-C(14)	1.50(2)	C(33)—O(3)	1.34 (2)
C(13)-C(18)	1.53 (2)	C(33)=O(4)	1.27 (2)
C(14)-C(15)	1-52 (2)	C(33)C(34)	1.42 (3)
C(15)-C(16)	1.56 (2)	C(35)—O(5)	1.34 (2)
C(16)-C(17)	1.52 (2)	C(35)=O(6)	1.21 (2)
C(17)-C(18)	1.54 (2)	C(35)-C(36)	1.45 (3)
C(17)-C(22)	1.58 (2)	C(37)—O(7)	1.31 (2)
C(18)-C(19)	1.53 (2)	C(37) = O(8)	1.30(3)
C(19)-C(20)	1.54 (2)	C(37)-C(38)	1.46 (3)
C(20)C(21)	1-56 (2)	C(39)-O(9)	1.40(2)
C(21)-C(22)	1.50 (2)	C(39)=O(10)	1.08(2)
O(21) O(22)	. 50 (2)	C(39)-C(40)	1.55 (3)
		((3), ((3),	
(iii) Glucopyrano	osyl moiety		
C(1')-O(11)	1.38(1)	C(7')-C(8')	1.54 (2)
C(1')-O(1')	1.42 (2)	C(7")O(2")	1.38 (2)
C(1')-C(2')	1.51 (2)	C(7")=O(3")	1.16 (2)
C(2')-O(4')	1.42(1)	C(7")–C(8")	1.56 (3)
C(2')-C(3')	1.55 (2)	C(8')-C(11')	1.53 (3)
C(3')-O(2'')	1.45 (1)	C(8')=C(9')	1.38 (3)
C(3')-C(4')	1.48 (2)	C(8'')-C(11'')	1.56 (3)
C(4')-O(2')	· 1·48 (1)	C(8'')=C(9'')	1.15 (3)
C(4')C(5')	1.57 (2)	C(9')-C(10')	1.53 (2)
C(5')-O(1')	1.41 (1)	C(9'')-C(10'')	1.53 (2)
C(5')C(6')	1.54 (2)	C(12')-O(4')	1.34(2)
C(7')-O(2')	1.34 (2)	C(12')=O(5')	1.21 (2)
C(7')=O(3')	1.20 (2)	C(12')-C(13')	1.52 (3)
	. ~~ (~)	/ /	

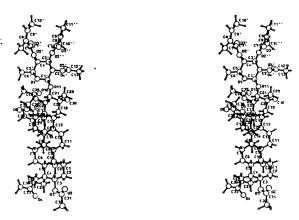


Fig. 2. Stereoscopic drawing of the molecule.

Table 3. Bond angles (°) with standard deviations in parentheses

C(1)=C(1)=C(10) C(1)=C(2)=C(3)=C(4) C(2)=C(3)=C(4) C(2)=C(3)=C(4) C(2)=C(3)=C(4) C(2)=C(3)=C(4) C(2)=C(3)=C(4) C(3)=C(4) C(3)=C(4) C(3)=C(4) C(3)=C(4) C(3)=C(4) C(3)=C(4) C(3)=C(4) C(3)=C(4) C(3)=C(4)-C(2) C(4)=C(2) C(4)=C(3) C(3)=C(4) C(4)=C(4)=C(4) C(4)=C(4)=C(4)=C(4)=C(4)=C(4)=C(4)=C(4)=	(i) Triterpene nucleus			
C(1)-C(2)-C(3)	C(2)-C(1)-C(10)	111 (1)	C(12)=C(13)C(18)	115 (1)
C(2)=C(3)=C(4)	C(1)-C(2)-C(3)		C(14)-C(13)C(18)	124 (1)
C(2)—C(3)—O(1) 107 (1) C(8)—C(14)—C(15) 110 (C(3)—C(4)—C(5) 106 (1) C(3)—C(4)—C(27) 112 (C(3)—C(4)—C(23) 109 (1) C(3)—C(4)—C(27) 110 (C(3)—C(4)—C(23) 109 (1) C(3)—C(4)—C(27) 109 (C(3)—C(4)—C(23) 107 (1) C(13)—C(14)—C(27) 109 (C(5)—C(4)—C(23) 107 (1) C(14)—C(15)—C(16)—118 (C(5)—C(4)—C(23) 107 (1) C(14)—C(15)—C(16)—C(17) 113 (C(2))—C(4)—C(24) 108 (1) C(15)—C(16)—C(16)—O(5) 108 (C(4)—C(5)—C(6) 117 (1) C(15)—C(16)—C(5) 108 (C(4)—C(5)—C(6) 117 (1) C(17)—C(16)—O(5) 107 (C(4)—C(5)—C(10) 108 (1) C(16)—C(17)—C(22) 113 (C(5)—C(6)—C(7)—C(8) 114 (1) C(16)—C(17)—C(22) 113 (C(5)—C(6)—C(7)—C(8) 114 (1) C(18)—C(17)—C(28) 107 (C(6)—C(7)—C(8)—10 (1) C(18)—C(17)—C(28) 107 (C(7)—C(8)—C(9)—(10) 109 (1) C(18)—C(17)—C(28) 109 (C(7)—C(8)—C(26) 110 (1) C(13)—C(18)—C(17)—C(28) 109 (C(7)—C(8)—C(26) 110 (1) C(13)—C(18)—C(17)—112 (C(9)—C(8)—C(26) 110 (1) C(13)—C(18)—C(17)—112 (C(9)—C(8)—C(26) 110 (1) C(13)—C(18)—C(17)—112 (C(9)—C(8)—C(26) 110 (1) C(13)—C(18)—C(19)—112 (C(9)—C(8)—C(26) 110 (1) C(18)—C(19)—C(20)—C(21) 104 (C(8)—C(9)—C(11) 111 (1) C(19)—C(20)—C(21) 104 (C(8)—C(9)—C(11) 111 (1) C(19)—C(20)—C(21) 104 (C(1)—C(9)—C(11) 112 (1) C(19)—C(20)—C(21) 104 (C(1)—C(10)—C(5) 108 (1) C(1)—C(10)—C(5) 108 (1) C(1)—C(10)—C(25) 107 (1) C(2)—C(20)—C(29) 110 (C(1)—C(10)—C(5) 108 (1) C(21)—C(20)—C(29) 110 (C(1)—C(10)—C(5) 108 (1) C(21)—C(20)—C(29) 110 (C(1)—C(10)—C(5) 115 (1) C(21)—C(20)—C(21)—O(11) 106 (C(9)—C(11)—C(12)—C(13) 125 (1) C(17)—C(22)—O(9) 105 (C(1)—C(12)—C(13) 125 (1) C(17)—C(22)—O(9) 105 (C(1)—C(1)—C(1)) 108 (1) C(2)—C(21)—C(11) 106 (22)—C(21)—C(11) 106 (22)—C(21)—C(11) 106 (22)—C(21)—C(11) 106 (22)—C(21)—C(11) 106 (22)—C(23)—C(14) 121 (1) C(11)—C(12)—C(13) 125 (1) C(17)—C(22)—O(9) 105 (C(1)—C(1)) 106 (22)—C(23)—106 (23)—C(24) 123 (1) O(1)—C(17)—C(18)—O(17)—C(18)—O(17)—O(17)—O(17) 108 (1) C(17)—C(18)—O(17)—O(17)—O(17) 109 (1) O(2)—C(17)—C(18) 119 (1				
C(a)—C(3)—C(4)—C(5)			C(0) C(14) C(15)	
$\begin{array}{c} C(3) - C(4) - C(5) \\ C(3) - C(4) - C(23) \\ C(5) - C(4) - C(24) \\ C(5) - C(4) - C(24) \\ C(5) - C(4) - C(24) \\ C(6) - C(17) \\ C(15) - C(16) - C(16) \\ C(17) - C(18) \\ C(4) - C(5) - C(16) \\ C(4) - C(5) - C(10) \\ C(4) - C(5) - C(10) \\ C(6) - C(7) - C(8) \\ C(9) - C(9) \\ C(9) - C(9) \\ C(9) - C(26) \\ C(11) \\ C(11) - C(26) \\ C(11) - C(27) \\ C(12) - C(28) \\ C(12) - C(26) \\ C(11) \\ C(11) - C(28) \\ C(12) - C(26) \\ C(11) \\ C(11) - C(18) - C(11) \\ C(11) - C(12) \\ C(11) \\ C(11) - C(12) \\ C(11) - C(12) \\ C(11) \\ C(11) \\ C(12) - C(13) \\ C(11) \\ C(12) - C(13) \\ C(11) \\ C(12) - C(12) \\ C(13) \\ C(11) \\ C(12) \\ C(13) \\ C(11) \\ C(12) \\ C(11) \\ C(12) \\ C(11) \\ C(12) \\ C(12) \\ C(13) \\ C(11) \\ C(12) \\ C(11) \\ C(12) \\ C(11) \\ C(12) \\ C(12) \\ C(11) \\ C(12) \\ C(11) \\ C(12) \\ C(12) \\ C$				
C(3)-C(4)-C(23) 108 (1) C(13)-C(14)-C(27) 109 (15)-C(14)-C(24) 117 (1) C(15)-C(16)-C(15) 118 (15)-C(16)-C(16)-C(17) 113 (15)-C(16)-C(16)-C(17) 113 (17)-C(16)-C(16)-C(19) 118 (17)-C(16)-C(16)-C(19) 118 (17)-C(16)-C(16)-C(17)-C(18) 119 (17)-C(16)-C(17)-C(18) 119 (17)-C(18)-C(17)-C(18) 119 (17)-C(18)-C(17)-C(18) 119 (17)-C(18)-C(18)-C(17)-C(18)-C(19			C(8)-C(14)-C(21)	
C(3)-C(4)-C(23) 108 (1) C(13)-C(14)-C(27) 109 (15)-C(14)-C(24) 117 (1) C(15)-C(16)-C(15) 118 (15)-C(16)-C(16)-C(17) 113 (15)-C(16)-C(16)-C(17) 113 (17)-C(16)-C(16)-C(19) 118 (17)-C(16)-C(16)-C(19) 118 (17)-C(16)-C(16)-C(17)-C(18) 119 (17)-C(16)-C(17)-C(18) 119 (17)-C(18)-C(17)-C(18) 119 (17)-C(18)-C(17)-C(18) 119 (17)-C(18)-C(18)-C(17)-C(18)-C(19			C(13)-C(14)-C(15)	110 (1)
C(3)-C(4)-C(23) 108 (1) C(13)-C(14)-C(27) 109 (15)-C(14)-C(24) 117 (1) C(15)-C(16)-C(15) 118 (15)-C(16)-C(16)-C(17) 113 (15)-C(16)-C(16)-C(17) 113 (17)-C(16)-C(16)-C(19) 118 (17)-C(16)-C(16)-C(19) 118 (17)-C(16)-C(16)-C(17)-C(18) 119 (17)-C(16)-C(17)-C(18) 119 (17)-C(18)-C(17)-C(18) 119 (17)-C(18)-C(17)-C(18) 119 (17)-C(18)-C(18)-C(17)-C(18)-C(19	C(3)-C(4)-C(23)	109 (1)	C(13)-C(14)-C(27)	106 (1)
$\begin{array}{c} C(s)-C(4)-C(23) & 107 (1) & C(14)-C(15)-C(16) & 118 (15)-C(4)-C(24) & 117 (1) & C(15)-C(16)-C(17) & 113 (15)-C(4)-C(24) & 108 (1) & C(15)-C(16)-C(15) & 108 (16)-C(16)-C(16)-C(16) & 107 (16)-C(16)-C(16) & 107 (16)-C(16)-C(16) & 107 (16)-C(16)-C(16) & 107 (16)-C(16)-C(16) & 107 (16)-C(17)-C(18) & 107 (16)-C(17)-C(28) & 107 (17)-C(18)-C(14) & 110 (1) & C(18)-C(17)-C(28) & 105 (17)-C(18)-C(14) & 110 (1) & C(12)-C(11)-C(28) & 105 (17)-C(18)-C(19) & 112 (17)-C(18)-C(19)-C(19) & 113 (11) & C(19)-C(20)-C(20) & 115 (18)-C(19)-C(20)-C(20) & 115 (18)-C(19)-C(20)-C(21) & 104 (11) & C(19)-C(20)-C(21) & 104 (11) & C(19)-C(20)-C(29) & 108 (11) & C(10)-C(10)-C(19) & 104 (11) & C(21)-C(20)-C(29) & 106 (11)-C(10)-C(12) & 107 (11) & C(29)-C(21)-C(11) & 108 (11)-C(10)-C(12) & 115 (11) & C(29)-C(21)-C(21) & 112 (11) & C(19)-C(10)-C(22) & 115 (11) & C(29)-C(21)-O(11) & 108 (19)-C(10)-C(25) & 115 (11) & C(29)-C(21)-O(11) & 108 (19)-C(20)-C(23) & 115 (11) & C(29)-C(21)-O(11) & 108 (19)-C(20)-C(21)-O(11) & 108 (19)-C($		108 (1)	C(15)-C(14)-C(27)	109 (1)
$\begin{array}{c} C(s)-C(a)-C(2a) & 117 (1) \\ C(23)-C(4)-C(24) & 108 (1) \\ C(4)-C(5)-C(6) & 117 (1) \\ C(4)-C(5)-C(6) & 117 (1) \\ C(5)-C(6)-C(10) & 116 (1) \\ C(6)-C(5)-C(10) & 108 (1) \\ C(5)-C(6)-C(7) & 110 (1) \\ C(5)-C(6)-C(7) & 110 (1) \\ C(6)-C(7)-C(8) & 114 (1) \\ C(7)-C(8)-C(9) & 109 (1) \\ C(7)-C(8)-C(10) & 108 (1) \\ C(7)-C(8)-C(14) & 110 (1) \\ C(7)-C(8)-C(14) & 110 (1) \\ C(9)-C(8)-C(14) & 110 (1) \\ C(9)-C(8)-C(14) & 110 (1) \\ C(9)-C(8)-C(14) & 110 (1) \\ C(9)-C(8)-C(26) & 110 (1) \\ C(9)-C(8)-C(26) & 110 (1) \\ C(9)-C(8)-C(26) & 111 (1) \\ C(9)-C(8)-C(26) & 111 (1) \\ C(9)-C(8)-C(20) & 111 (1) \\ C(9)-C(8)-C(20) & 111 (1) \\ C(9)-C(8)-C(20) & 115 (1) \\ C(10)-C(9)-C(11) & 111 (1) \\ C(10)-C(9)-C(11) & 112 (1) \\ C(10)-C(9)-C(11) & 112 (1) \\ C(10)-C(9)-C(11) & 112 (1) \\ C(10)-C(9)-C(10) & 104 (1) \\ C(1)-C(10)-C(9) & 104 (1) \\ C(1)-C(10)-C(25) & 107 (1) \\ C(2)-C(20)-C(20) & 110 \\ C(3)-C(10)-C(9) & 106 (1) \\ C(3)-C(10)-C(25) & 116 (1) \\ C(9)-C(10)-C(25) & 115 (1) \\ C(9)-C(10)-C(25) & 115 (1) \\ C(1)-C(10)-C(25) & 115 (1) \\ C(20)-C(21)-C(12) \\ C(1)-C(1)-C(12)-C(13) & 125 (1) \\ C(1)-C(12)-C(13) & 125 (1) \\ C(1)-C(12)-C(13) & 125 (1) \\ C(11)-C(12)-C(13) & 126 (1) \\ C(11)-C(12)-C(13) & 106 (1) \\ C(2)-C(3)-C(4) & 106 (3) (9) \\ C(2)-C(3)-C(4) & 106 (3) (9) \\ C(2)-C(3)-C(4) $				118 (1)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(S)-C(A)-C(2A)			113 (1)
$\begin{array}{c} C(4)-C(5)-C(6) & 117 (1) & C(17)-C(16)-O(5) & 107 \\ C(4)-C(5)-C(10) & 116 (1) & C(16)-C(17)-C(18) & 114 \\ C(5)-C(5)-C(10) & 108 (1) & C(16)-C(17)-C(28) & 107 \\ C(6)-C(7)-C(8) & 114 (1) & C(18)-C(17)-C(22) & 110 \\ C(5)-C(6)-C(7) & 110 (1) & C(18)-C(17)-C(22) & 110 \\ C(7)-C(8)-C(9) & 109 (1) & C(18)-C(17)-C(28) & 109 \\ C(7)-C(8)-C(14) & 110 (1) & C(22)-C(17)-C(28) & 109 \\ C(7)-C(8)-C(14) & 110 (1) & C(13)-C(18)-C(17) & 112 \\ C(9)-C(8)-C(14) & 107 3 (9) & C(13)-C(18)-C(19) & 112 \\ C(9)-C(8)-C(26) & 111 (1) & C(17)-C(18)-C(19) & 112 \\ C(9)-C(8)-C(26) & 110 (1) & C(18)-C(19)-C(20) & 115 \\ C(14)-C(8)-C(26) & 110 (1) & C(18)-C(19)-C(20) & 115 \\ C(8)-C(9)-C(11) & 115 (1) & C(19)-C(20)-C(29) & 108 \\ C(10)-C(9)-C(11) & 112 (1) & C(19)-C(20)-C(29) & 108 \\ C(10)-C(9)-C(11) & 112 (1) & C(19)-C(20)-C(29) & 108 \\ C(1)-C(10)-C(5) & 108 (1) & C(21)-C(20)-C(29) & 110 \\ C(1)-C(10)-C(9) & 104 (1) & C(21)-C(20)-C(30) & 114 \\ C(1)-C(10)-C(9) & 104 (1) & C(21)-C(20)-C(30) & 114 \\ C(5)-C(10)-C(9) & 106 (1) & C(20)-C(21)-C(12) \\ C(5)-C(10)-C(9) & 106 (1) & C(20)-C(21)-C(12) \\ C(5)-C(10)-C(25) & 115 (1) & C(20)-C(21)-C(11) & 108 \\ C(9)-C(11)-C(12) & 115 (1) & C(22)-C(21)-O(11) & 106 \\ C(9)-C(11)-C(12) & 115 (1) & C(17)-C(22)-C(21) & 117 \\ C(11)-C(12)-C(13) & 125 (1) & C(17)-C(22)-C(21) & 117 \\ C(11)-C(12)-C(13) & 125 (1) & C(17)-C(22)-C(21) & 107 \\ C(12)-C(13)-C(14) & 121 (1) & C(21)-C(20)-C(38) & 129 \\ C(21)-O(11)-C(17) & 116 (3 (9) & O(6)-C(35)-C(36) & 129 \\ C(21)-O(11)-C(17) & 116 (3 (9) & O(6)-C(35)-C(36) & 129 \\ C(21)-C(13)-C(32) & 128 (2) & O(9)-C(39)-C(40) & 104 \\ O(1)-C(3)-C(3) & 109 (1) & O(7)-C(37)-C(38) & 113 \\ C(11)-C(12)-C(2) & 110 (1) & O(7)-C(37)-C(38) & 113 \\ C(11)-C(17)-C(2) & 110 (1) & O(7)-C(37)-C(8) & 109 \\ O(1)-C(3)-C(3) & 106 (1) & O(2)-C(7)-O(3) & 119 \\ O(1)-C(1)-C(1) & 108 (1) & O(2)-C(7)-O(3) & 119 \\ O(1)-C(1)-C(1) & 108 (1) & O(2)-C(7)-O(3) & 119 \\ O(1)-C(1)-C(1) & 108 (1) & O(2)-C(7)-O(3) & 119 \\ O(1)-C(1)-C(1) & 109 (1) & O(2)-C(7)-O(3) & 119 \\ O(1)-C(1)-C(1) & 108 (1) & O(2)-C(7)-C(8) & 120 \\ O(1)-C(1$				108.0 (9)
$\begin{array}{c} C(4)-C(5)-C(10) & 116 (1) \\ C(6)-C(5)-C(10) & 108 (1) \\ C(6)-C(7)-C(10) & 108 (1) \\ C(5)-C(6)-C(7) & 110 (1) \\ C(6)-C(7)-C(22) & 113 \\ C(7)-C(8)-C(9) & 109 (1) \\ C(7)-C(8)-C(9) & 109 (1) \\ C(7)-C(8)-C(14) & 110 (1) \\ C(9)-C(8)-C(26) & 110 (1) \\ C(9)-C(8)-C(26) & 110 (1) \\ C(9)-C(8)-C(26) & 111 (1) \\ C(13)-C(18)-C(17) & 112 \\ C(9)-C(8)-C(26) & 111 (1) \\ C(13)-C(18)-C(19) & 113 \\ C(14)-C(8)-C(26) & 110 (1) \\ C(19)-C(9)-C(10) & 115 (1) \\ C(19)-C(9)-C(20) & 113 \\ C(10)-C(9)-C(11) & 111 (1) \\ C(19)-C(20)-C(29) & 108 \\ C(10)-C(9)-C(11) & 111 (1) \\ C(1)-C(10)-C(9) & 104 (1) \\ C(1)-C(10)-C(9) & 104 (1) \\ C(1)-C(10)-C(9) & 104 (1) \\ C(1)-C(10)-C(25) & 107 (1) \\ C(29)-C(20)-C(30) & 114 \\ C(1)-C(10)-C(25) & 115 (1) \\ C(9)-C(10)-C(25) & 115 (1) \\ C(9)-C(10)-C(25) & 115 (1) \\ C(9)-C(10)-C(25) & 115 (1) \\ C(12)-C(12)-C(12) & 114 (1) \\ C(11)-C(12)-C(13) & 125 (1) \\ C(11)-C(12)-C(13) & 125 (1) \\ C(11)-C(12)-C(13) & 125 (1) \\ C(11)-C(12)-C(29)-C(10) & 105 \\ C(28)-O(7)-C(28) & 119 (1) \\ C(21)-C(22)-O(9) & 105 \\ C(28)-O(7)-C(28) & 119 (1) \\ C(1)-C(33)-C(34) & 131 \\ C(10)-C(33)-C(34) & 121 (1) \\ C(11)-C(13)-C(31) & 109 (1) \\ C(17)-C(28)-O(7) & 109 (1) \\ C(17)-C(28)-O(7) & 109 (1) \\ C(17)-C(29)-C(37) & 110 (1) \\ C(17)-C(29)-C(37) & 110 (1) \\ C(17)-C(29)-C(37) & 110 (1) \\ C(17)-C(27)-C(47) & 110 (1) \\ C(27)-C(17)-C(8) & 113 (1) \\ C(17)-C(27)-C(8) & 110 (1) \\ C(17)-C(27)-C(8) & 110 (1) \\ C(17)-C(17)-C(17) & 108 (1) \\ C(17)-C(17)-C(18) & 110 (1) \\ C(17)-C(18)-C(17) & 108 (1) \\ $				
$\begin{array}{c} C(6)-C(5)-C(10) & 108 (1) & C(16)-C(17)-C(22) & 113 (10) \\ C(5)-C(6)-C(7)-C(8) & 114 (1) & C(18)-C(17)-C(28) & 107 (10) \\ C(7)-C(8)-C(9) & 109 (1) & C(18)-C(17)-C(28) & 105 (10) \\ C(7)-C(8)-C(9) & 109 (1) & C(18)-C(17)-C(28) & 109 (10) \\ C(7)-C(8)-C(26) & 110 (1) & C(22)-C(17)-C(28) & 109 (10) \\ C(9)-C(8)-C(14) & 107 -3 (9) & C(13)-C(18)-C(19) & 112 (10) \\ C(9)-C(8)-C(14) & 107 -3 (9) & C(13)-C(18)-C(19) & 112 (10) \\ C(9)-C(8)-C(26) & 111 (1) & C(17)-C(18)-C(19) & 112 (10) \\ C(9)-C(8)-C(26) & 111 (1) & C(18)-C(19)-C(20) & 115 (10) \\ C(8)-C(9)-C(10) & 115 (1) & C(19)-C(20)-C(21) & 104 (10) \\ C(8)-C(9)-C(11) & 111 (1) & C(19)-C(20)-C(20) & 108 (10) \\ C(10)-C(9)-C(11) & 112 (1) & C(19)-C(20)-C(29) & 108 (10) \\ C(1)-C(10)-C(5) & 108 (1) & C(21)-C(20)-C(29) & 110 (10) \\ C(1)-C(10)-C(25) & 107 (1) & C(29)-C(20)-C(30) & 114 (10) \\ C(1)-C(10)-C(25) & 107 (1) & C(29)-C(20)-C(30) & 104 (10) \\ C(5)-C(10)-C(25) & 115 (1) & C(20)-C(21)-O(11) & 108 \\ C(9)-C(11)-C(12) & 114 (1) & C(17)-C(22)-C(21) & 117 (10) \\ C(9)-C(11)-C(12) & 114 (1) & C(17)-C(22)-O(9) & 105 (10) \\ C(11)-C(12)=C(13) & 125 (1) & C(17)-C(22)-O(9) & 105 (10) \\ C(12)=C(13)-C(14) & 121 (1) & C(21)-C(23)-C(34) & 131 (10) \\ C(10)-C(31)-C(28) & 119 (1) & O(5)-C(35)-C(36) & 111 (10) \\ C(17)-C(28)-O(7) & 109 (1) & O(7)-C(37)-C(8) & 129 (10) \\ O(1)-C(31)-C(32) & 106 (1) & O(9)-C(39)-C(40) & 104 (10) \\ O(3)-C(33)-C(34) & 114 (2) & O(7)-C(37)-C(8) & 129 (10) \\ C(17)-C(29)-C(47) & 100 (1) & O(7)-C(37)-C(8) & 129 (10) \\ C(17)-C(27)-C(47) & 110 (1) & O(3)-C(77)-C(87) & 109 (10) \\ C(17)-C(27)-C(47) & 110 (1) & O(3)-C(77)-C(87) & 110 (10) \\ C(17)-C(27)-C(47) & 110 (1) & O(3)-C(77)-C(87) & 120 (10) \\ C(27)-C(17)-O(17) & 108 (1) & C(17)-C(87)-C(117) & 118 (10) \\ C(17)-C(27)-C(47) & 110 (1) & O(3)-C(77)-C(87) & 120 (10) \\ C(27)-C(47)-C(57) & 110 (1) & O(37)-C(77)-C(87) & 120 (10) \\ C(47)-C(57)-O(17) & 108 (1) & C(87)-C(87)-C(117) & 130 (10) \\ C(47)-C(57)-O(17) & 108 (1) & C(87)-C(87)-C(117) & 130 (10) \\ C(47)-C(57)-O(17) & 108 (1) & C(87)-C(87)-C(117) & 130 (10) \\ C(47)$			C(17)-C(10)-O(3)	
$\begin{array}{c} C(5)-C(6)-C(7) \\ C(6)-C(7)-C(8) \\ C(7)-C(8) \\ C(7)-C(8)-C(9) \\ C(7)-C(8)-C(9) \\ C(7)-C(8)-C(14) \\ C(9)-C(8)-C(14) \\ C(9)-C(10) \\ C(9)-C(8)-C(14) \\ C(9)-C(8)-C(14) \\ C(9)-C(10) \\ C(9)-C(8)-C(26) \\ C(11) \\ C(11) \\ C(11)-C(19)-C(20) \\ C(11) \\ C(11) \\ C(11)-C(11) \\ C(11) \\ C(11)-C(10)-C(11) \\ C(11) \\ C(11)-C(10)-C(11) \\ C(11) \\ C(11)-C(10)-C(11) \\ C(11) \\ C(11)-C(10)-C(12) \\ C(11) \\ C(11)-C(10)-C(25) \\ C(11) \\ C(11)-C(10)-C(25) \\ C(11) \\ C(11)-C(12)-C(13) \\ C(11)-C(12)-C(23) \\ C(11)-C(12)-C(23) \\ C(11)-C(12)-C(23) \\ C(11)-C(12)-C(23) \\ C(11)-C(12)-C(23) \\ C(11)-C(12)-C(23) \\ C(21)-C(13)-C(14) \\ C(11)-C(12)-C(22)-C(21) \\ C(11)-C(12)-C(23) \\ C(21)-C(13)-C(24) \\ C(21)-C(21)-C(22)-C(21) \\ C(21)-C(21)-C(23) \\ C(21)-C(11)-C(12) \\ C(11)-C(12)-C(23) \\ C(21)-C(12)-C(23) \\ C(21)-C(11)-C(12) \\ C(11)-C(12)-C(23) \\ C(21)-C(11)-C(13) \\ C(11)-C(12)-C(23) \\ C(21)-C(11)-C(23) \\ C(21)-C(11)-C(23) \\ C(21)-C(11)-C(12) \\ C(11)-C(12)-C(23) \\ C(21)-C(11)-C(13) \\ C(11)-C(12)-C(23) \\ C(21)-C(11)-C(13) \\ C(11)-C(12)-C(23) \\ C(21)-C(11)-C(12) \\ C(11)-C(12)-C(23) \\ C(21)-C(11)-C(23) \\ C(21)-C(11)-C(21) \\ C(11)-C(12)-C(23) \\ C(21)-C(11)-C(21) \\ C(11)-C(12)-C(21) \\ C(21)-C(21)-C(21) \\ C(2$			C(16)-C(17)-C(18)	
C(6)—C(7)—C(8) C(7)—C(8)—C(9) D(9) D(9) D(10) C(18)—C(17)—C(22) D(17)—C(28) D(9) C(7)—C(8)—C(14) D(10) C(7)—C(8)—C(26) D(10) C(7)—C(8)—C(26) D(10) C(9)—C(8)—C(14) D(7)—C(8)—C(26) D(11) D(11) C(13)—C(18)—C(17) D(12)—C(19) D(13)—C(18)—C(17) D(12)—C(20)—C(20) D(13)—C(19)—C(20) D(13)—C(19)—C(20) D(13)—C(19)—C(20)—C(21) D(10)—C(9)—C(10) D(11) D(11) D(11)—C(20)—C(21) D(10)—C(20)—C(21) D(10)—C(20)—C(21) D(10)—C(20)—C(21) D(10)—C(20)—C(20) D(10)—C(20)—D(20) D(10)—C(20)—C(20) D(20)—C(20)—C(20) D(20)—C(20)—C(20)—D(20) D(20)—C(20)—D(20) D(20)—C(20)—D(20) D(20)—C(20)—D(20) D(20)—C(20)—D(20) D(20)—C(20)—D(20) D(20)—D(20)—D(20) D(20)—C(20)—D(20)—D(20) D(20)—C(20)—D(20) D(20)—D(20)—D(20) D(20)—D(20)—D(20)—D(20) D(20)—D(20)—D(20)—D(20) D(20)—D(20)—D(20)—D(20)—D(20) D(20)—D(113 (1)
C(7)—C(8)—C(9) 109 (1) C(18)—C(17)—C(28) 109 (1) C(7)—C(8)—C(14) 110 (1) C(22)—C(17)—C(28) 109 (1) C(7)—C(8)—C(14) 110 (1) C(13)—C(18)—C(17) 112 (12)—C(9)—C(8)—C(14) 107-3 (9) C(13)—C(18)—C(19) 113 (14)—C(8)—C(26) 111 (1) C(17)—C(18)—C(19)—C(20)—C(21) 104 (17)—C(20)—C(21) 111 (1) C(19)—C(20)—C(21) 104 (17)—C(20)—C(21) 111 (1) C(19)—C(20)—C(21) 104 (17)—C(20)—C(21) 111 (1) C(19)—C(20)—C(21) 104 (17)—C(20)—C(21) 111 (1) C(19)—C(20)—C(20)—C(21) 104 (17)—C(20)—C(21) 111 (1) C(19)—C(20)—C(20)—C(20) 112 (17)—C(10)—C(5) 108 (1) C(21)—C(20)—C(20)—C(30) 112 (17)—C(10)—C(9) 104 (1) C(21)—C(20)—C(23) 114 (1) C(1)—C(10)—C(9) 104 (1) C(21)—C(20)—C(30) 109 (17)—C(10)—C(25) 115 (1) C(20)—C(21)—O(11) 108 (1) C(9)—C(10)—C(25) 115 (1) C(20)—C(21)—O(11) 108 (1) C(9)—C(10)—C(25) 115 (1) C(22)—C(21)—O(11) 108 (1) C(11)—C(12)—C(13) 125 (1) C(17)—C(22)—O(9) 107 (12)—C(13)—C(14) 121 (1) C(21)—C(22)—O(9) 105 (12)—C(21)—O(11)—C(17) 116-3 (9) O(6)—C(35)—C(36) 129 (17)—C(28)—O(7)—O(3) 109 (1) O(7)—C(37)—C(38) 113 (1) C(10)—C(31)—C(32)—D(8) 116 (17)—C(28)—O(7)—O(3) 109 (1) O(7)—C(37)—C(38) 129 (17)—C(28)—O(7)—O(3) 109 (1) O(7)—C(37)—C(38) 123 (1) O(1)—C(31)—C(32)—D(32) 128 (2) O(9)—C(39)—C(40) 104 (1) O(3)—C(33)—C(34) 114 (2) (10)—C(31)—C(32)—D(34) 114 (2) (10)—C(31)—C(32)—D(34) 114 (2) (10)—C(33)—C(34) 114 (2) (10)—C(33)—C(34) 114 (2) (10)—C(33)—C(34) 114 (2) (10)—C(37)—C(38) 100 (1) O(7)—C(37)—C(38) 113 (10)—C(77)—C(87)—D(18) 113 (10)—C(77)—C(87)—D(18) 113 (10)—C(77)—C(87)—D(18) 113 (10)—C(77)—C(87)—D(18) 114 (2) (10)—C(79)—C(87)—C(87)—D(17) 109 (1) O(7)—C(87)—C(87)—C(87)—D(17) 109 (1) O(7)—C(87)—C(87)—C(87)—C(17)—O(87) 112 (10) (10)—C(17)—C(17)—D(17) 109 (1) O(27)—C(17)—O(87) 126 (17)—C(18)—C(17)—D(18) 113 (10) (10)—C(17)—C(18)—C(17)—D(18) 113 (10) (10)—C(17)—C(18)—D(18) 113 (10) (10)—C(17)—C(18)—D(18) 113 (10) (10)—C(17)—C(18)—D(18) 113 (10) (10)—C(17)—C(18)—D(18) 113 (10) (10)—C(17)—D(C(5)-C(6)-C(7)		C(16)-C(17)-C(28)	107 (1)
C(7)—C(8)—C(9) 109 (1) C(18)—C(17)—C(28) 109 (1) C(7)—C(8)—C(14) 110 (1) C(22)—C(17)—C(28) 109 (1) C(7)—C(8)—C(14) 110 (1) C(13)—C(18)—C(17) 112 (12)—C(9)—C(8)—C(14) 107-3 (9) C(13)—C(18)—C(19) 113 (14)—C(8)—C(26) 111 (1) C(17)—C(18)—C(19)—C(20)—C(21) 104 (17)—C(20)—C(21) 111 (1) C(19)—C(20)—C(21) 104 (17)—C(20)—C(21) 111 (1) C(19)—C(20)—C(21) 104 (17)—C(20)—C(21) 111 (1) C(19)—C(20)—C(21) 104 (17)—C(20)—C(21) 111 (1) C(19)—C(20)—C(20)—C(21) 104 (17)—C(20)—C(21) 111 (1) C(19)—C(20)—C(20)—C(20) 112 (17)—C(10)—C(5) 108 (1) C(21)—C(20)—C(20)—C(30) 112 (17)—C(10)—C(9) 104 (1) C(21)—C(20)—C(23) 114 (1) C(1)—C(10)—C(9) 104 (1) C(21)—C(20)—C(30) 109 (17)—C(10)—C(25) 115 (1) C(20)—C(21)—O(11) 108 (1) C(9)—C(10)—C(25) 115 (1) C(20)—C(21)—O(11) 108 (1) C(9)—C(10)—C(25) 115 (1) C(22)—C(21)—O(11) 108 (1) C(11)—C(12)—C(13) 125 (1) C(17)—C(22)—O(9) 107 (12)—C(13)—C(14) 121 (1) C(21)—C(22)—O(9) 105 (12)—C(21)—O(11)—C(17) 116-3 (9) O(6)—C(35)—C(36) 129 (17)—C(28)—O(7)—O(3) 109 (1) O(7)—C(37)—C(38) 113 (1) C(10)—C(31)—C(32)—D(8) 116 (17)—C(28)—O(7)—O(3) 109 (1) O(7)—C(37)—C(38) 129 (17)—C(28)—O(7)—O(3) 109 (1) O(7)—C(37)—C(38) 123 (1) O(1)—C(31)—C(32)—D(32) 128 (2) O(9)—C(39)—C(40) 104 (1) O(3)—C(33)—C(34) 114 (2) (10)—C(31)—C(32)—D(34) 114 (2) (10)—C(31)—C(32)—D(34) 114 (2) (10)—C(33)—C(34) 114 (2) (10)—C(33)—C(34) 114 (2) (10)—C(33)—C(34) 114 (2) (10)—C(37)—C(38) 100 (1) O(7)—C(37)—C(38) 113 (10)—C(77)—C(87)—D(18) 113 (10)—C(77)—C(87)—D(18) 113 (10)—C(77)—C(87)—D(18) 113 (10)—C(77)—C(87)—D(18) 114 (2) (10)—C(79)—C(87)—C(87)—D(17) 109 (1) O(7)—C(87)—C(87)—C(87)—D(17) 109 (1) O(7)—C(87)—C(87)—C(87)—C(17)—O(87) 112 (10) (10)—C(17)—C(17)—D(17) 109 (1) O(27)—C(17)—O(87) 126 (17)—C(18)—C(17)—D(18) 113 (10) (10)—C(17)—C(18)—C(17)—D(18) 113 (10) (10)—C(17)—C(18)—D(18) 113 (10) (10)—C(17)—C(18)—D(18) 113 (10) (10)—C(17)—C(18)—D(18) 113 (10) (10)—C(17)—C(18)—D(18) 113 (10) (10)—C(17)—D(C(6)-C(7)-C(8)	114 (1)	C(18)C(17)C(22)	110(1)
C(7)-C(8)-C(14)	C(7)-C(8)-C(9)	109 (1)	C(18)C(17)C(28)	105 (1)
$\begin{array}{c} C(7)-C(8)-C(26) & 110 (1) & C(13)-C(18)-C(17) & 112 (12) \\ C(9)-C(8)-C(14) & 107 \cdot 3 (9) & C(13)-C(18)-C(19) & 112 (12) \\ C(9)-C(8)-C(26) & 111 (1) & C(17)-C(18)-C(19) & 113 (12) \\ C(14)-C(8)-C(26) & 110 (1) & C(18)-C(20)-C(20) & 104 (12) \\ C(8)-C(9)-C(10) & 115 (1) & C(19)-C(20)-C(20) & 104 (12) \\ C(8)-C(9)-C(11) & 111 (1) & C(19)-C(20)-C(20) & 108 (12) \\ C(10)-C(9)-C(11) & 112 (1) & C(19)-C(20)-C(30) & 112 (12) \\ C(1)-C(10)-C(5) & 108 (1) & C(21)-C(20)-C(30) & 114 (12) \\ C(1)-C(10)-C(29) & 104 (1) & C(21)-C(20)-C(30) & 114 (12) \\ C(1)-C(10)-C(29) & 104 (1) & C(21)-C(20)-C(30) & 114 (12) \\ C(1)-C(10)-C(25) & 107 (1) & C(29)-C(21)-C(20) & 109 (12) \\ C(5)-C(10)-C(25) & 116 (1) & C(20)-C(21)-C(21) & 109 (12) \\ C(5)-C(10)-C(25) & 115 (1) & C(20)-C(21)-O(11) & 108 \\ C(9)-C(11)-C(12) & 115 (1) & C(22)-C(21)-O(11) & 106 \\ C(9)-C(11)-C(12) & 115 (1) & C(21)-C(22)-C(21) & 117 (12) \\ C(11)-C(12)=C(13) & 125 (1) & C(17)-C(22)-O(9) & 107 \\ C(12)=C(13)-C(14) & 121 (1) & C(21)-C(22)-O(9) & 105 \\ \hline (ii) Extranuclear & C(24)-O(3)-C(24) & 123 (1) & O(4)=C(33)-C(34) & 131 (16) \\ C(21)-O(1)-C(1)' & 116 \cdot 3 (9) & O(6)=C(35)-C(36) & 129 \\ C(3)-C(3)-O(7)-C(28) & 119 (1) & O(5)-C(35)-C(36) & 129 \\ C(4)-C(24)-O(3) & 109 (1) & O(7)-C(37)-O(8) & 116 (16) \\ C(7)-C(28)-O(7) & 109 (1) & O(7)-C(37)-C(38) & 123 (16) \\ O(1)-C(31)=O(2) & 125 (2) & O(8)=C(37)-C(38) & 125 (16) \\ O(2)-C(31)=C(32) & 128 (2) & O(9)-C(39)-C(40) & 104 (16) \\ O(3)-C(33)-C(34) & 115 (2) & O(10)=C(39)-C(40) & 104 (16) \\ O(3)-C(33)-C(34) & 115 (2) & O(10)=C(39)-C(40) & 104 (16) \\ C(7)-C(7)-O(17) & 108 (1) & C(7)-C(8)-C(17) & 126 (17) \\ C(7)-C(7)-C(8)' & 100 (1) & O(7)-C(8)'-C(8)' & 109 (10) \\ C(7)-C(7)-O(17) & 108 (1) & C(7)-C(8)'-C(117) & 130 (10) \\ C(7)-C(7)-O(17) & 108 (1) & C(7)-C(8)'-C(117) & 130 (10) \\ C(7)-C(7)-O(17) & 108 (1) & C(8)-C(9)-C(117) & 125 (10) \\ C(4)-C(5)-O(17) & 108 (1) & C(8)-C(9)-C(107) & 126 (17) \\ C(4)-C(5)-O(17) & 108 (1) & C(8)-C(9)-C(107) & 125 (10) \\ C(4)-C(5)-O(17) & 108 (1) & C(8)-C(9)-C(107) & 125 (10) \\ C(4)-C(5)-O(17) & 108 (1) &$			C(22)-C(17)-C(28)	109 (1)
C(9)-C(8)-C(14) 107·3 (9) C(13)-C(18)-C(19) 112·0 (9)-C(8)-C(26) 111 (1) C(17)-C(18)-C(19) 113·0 (14)-C(8)-C(26) 110 (1) C(18)-C(19)-C(20) 115·1 (18)-C(19)-C(20)-C(21) 104·0 (18)-C(19)-C(20)-C(21) 104·0 (18)-C(19)-C(20)-C(21) 104·0 (18)-C(19)-C(20)-C(21) 104·0 (18)-C(19)-C(20)-C(21) 104·0 (19)-C(20)-C(21) 104·0 (19)-C(20)-C(21) 104·0 (19)-C(20)-C(21) 108·0 (19)-C(20)-C(21) 111·1 (1) C(19)-C(20)-C(29) 108·0 (10)-C(9)-C(11) 112 (1) C(19)-C(20)-C(20) 110·0 (10)-C(10)-C(9) 104·1 (1) C(21)-C(20)-C(30) 114·0 (10)-C(10)-C(25) 107·1 (1) C(29)-C(20)-C(30) 109·0 (10)-C(10)-C(25) 116·1 (1) C(20)-C(21)-C(21)-10·0 (10)-C(25) 115·1 (1) C(20)-C(21)-O(11) 108·0 (10)-C(10)-C(25) 115·1 (1) C(22)-C(21)-O(11) 108·0 (10)-C(10)-C(25) 115·1 (1) C(22)-C(21)-O(11) 108·0 (10)-C(10)-C(12) 114·1 (1) C(17)-C(22)-O(9) 107·0 (11)-C(12)-C(13)-C(14) 121·1 (1) C(21)-C(22)-O(9) 105·0 (10)-C(31)-C(31)-C(31)-C(31) 125·1 (1) C(21)-C(22)-O(9) 105·0 (10)-C(31)-C				112(1)
$\begin{array}{c} C(9)-C(8)-C(26) & 111 & (1) & C(17)-C(18)-C(19) & 113 \\ C(14)-C(8)-C(26) & 110 & (1) & C(18)-C(20)-C(21) & 104 \\ C(8)-C(9)-C(10) & 115 & (1) & C(19)-C(20)-C(21) & 104 \\ C(8)-C(9)-C(11) & 111 & (1) & C(19)-C(20)-C(29) & 108 \\ C(10)-C(9)-C(11) & 111 & (1) & C(19)-C(20)-C(29) & 108 \\ C(10)-C(10)-C(5) & 108 & (1) & C(21)-C(20)-C(30) & 112 \\ C(1)-C(10)-C(9) & 104 & (1) & C(21)-C(20)-C(30) & 114 \\ C(1)-C(10)-C(29) & 106 & (1) & C(29)-C(20)-C(30) & 109 \\ C(5)-C(10)-C(9) & 106 & (1) & C(29)-C(21)-C(21) & 112 \\ C(5)-C(10)-C(9) & 106 & (1) & C(20)-C(21)-C(21) & 112 \\ C(5)-C(10)-C(25) & 116 & (1) & C(20)-C(21)-C(11) & 108 \\ C(9)-C(11)-C(12) & 114 & (1) & C(17)-C(22)-C(21) & 117 \\ C(11)-C(12)=C(13) & 125 & (1) & C(17)-C(22)-C(21) & 117 \\ C(11)-C(12)=C(13) & 125 & (1) & C(17)-C(22)-O(9) & 107 \\ C(12)=C(13)-C(14) & 121 & (1) & C(21)-C(22)-O(9) & 105 \\ \hline (ii) Extranuclear & C(24)-O(3)-C(24) & 123 & (1) & O(4)=C(33)-C(34) & 131 \\ C(21)-O(11)-C(11) & 116 & 3(9) & O(6)=C(35)-C(36) & 120 \\ C(28)-O(7)-C(28) & 119 & (1) & O(5)-C(35)=C(36) & 120 \\ C(21)-O(11)-C(11) & 116 & 3(9) & O(6)-C(35)-C(36) & 120 \\ C(21)-C(21)-O(11)-C(11) & 116 & 3(9) & O(6)-C(35)-C(36) & 120 \\ C(17)-C(28)-O(7) & 109 & (1) & O(7)-C(37)-C(38) & 113 \\ O(1)-C(31)=O(2) & 106 & (2) & O(9)-C(39)-C(40) & 104 \\ O(3)-C(33)-C(32) & 106 & (2) & O(9)-C(39)-C(40) & 104 \\ O(3)-C(33)-C(34) & 115 & (2) & O(10)=C(39)-C(40) & 104 \\ O(3)-C(33)-C(34) & 116 & (2) & O(9)-C(39)-C(40) & 104 \\ O(3)-C(33)-C(44) & 110 & (1) & O(2^*)-C(7^*)-O(8^*) & 122 \\ C(1^*)-C(1^*)-O(1^*) & 108 & 8(9) & O(2^*)-C(7^*)-O(8^*) & 122 \\ C(1^*)-C(2^*)-C(4^*) & 110 & (1) & O(3^*)-C(7^*)-C(8^*) & 109 \\ C(2^*)-C(3^*)-O(2^*) & 104 & (1) & C(7^*)-C(8^*)-C(11^*) & 108 \\ C(2^*)-C(3^*)-O(2^*) & 104 & (1) & C(7^*)-C(8^*)-C(11^*) & 108 \\ C(2^*)-C(3^*)-O(2^*) & 104 & (1) & C(7^*)-C(8^*)-C(11^*) & 106 \\ C(3^*)-C(4^*)-C(5^*) & 104 & (1) & C(7^*)-C(8^*)-C(11^*) & 106 \\ C(4^*)-C(5^*)-O(1^*) & 108 & (1) & C(8^*)-C(9^*)-C(11^*) & 106 \\ C(4^*)-C(5^*)-O(1^*) & 108 & (1) & C(8^*)-C(9^*)-C(11^*) & 106 \\ C(4^*)-C(5^*)-O($				112 (1)
$\begin{array}{c} C(14)-C(8)-C(26) & 110 (1) & C(18)-C(19)-C(20) & 115 (1) \\ C(8)-C(9)-C(10) & 115 (1) & C(19)-C(20)-C(21) & 104 (1) \\ C(8)-C(9)-C(11) & 111 (1) & C(19)-C(20)-C(20) & 108 (1) \\ C(10)-C(9)-C(11) & 112 (1) & C(19)-C(20)-C(30) & 112 (1) \\ C(1)-C(10)-C(5) & 108 (1) & C(21)-C(20)-C(30) & 114 (1) \\ C(1)-C(10)-C(9) & 104 (1) & C(21)-C(20)-C(30) & 114 (1) \\ C(1)-C(10)-C(25) & 107 (1) & C(29)-C(20)-C(30) & 114 (1) \\ C(1)-C(10)-C(25) & 106 (1) & C(20)-C(21)-C(22) & 112 (1) \\ C(5)-C(10)-C(25) & 116 (1) & C(20)-C(21)-C(11) & 108 \\ C(9)-C(10)-C(25) & 115 (1) & C(22)-C(21)-O(11) & 106 \\ C(9)-C(10)-C(25) & 115 (1) & C(22)-C(21)-O(11) & 106 \\ C(9)-C(11)-C(12) & 114 (1) & C(17)-C(22)-C(21) & 117 (1) \\ C(11)-C(12)=C(13) & 125 (1) & C(17)-C(22)-O(9) & 107 \\ C(12)=C(13)-C(14) & 121 (1) & C(21)-C(22)-O(9) & 105 \\ \hline (ii) Extranuclear & & & & & & & & & \\ C(24)-O(3)-C(24) & 123 (1) & O(4)=C(33)-C(34) & 131 (1) \\ C(21)-O(11)-C(1') & 116 -3 (9) & O(6)=C(35)-C(36) & 129 \\ C(4)-C(24)-O(3) & 109 (1) & O(7)-C(37)-O(8) & 116 (1) \\ C(21)-O(11)-C(1') & 116 -3 (9) & O(6)=C(35)-C(36) & 129 \\ C(4)-C(24)-O(3) & 109 (1) & O(7)-C(37)-C(38) & 123 (1) \\ O(1)-C(31)=O(2) & 125 (2) & O(8)=C(37)-C(38) & 129 (1) \\ O(1)-C(31)=O(2) & 125 (2) & O(8)=C(37)-C(38) & 129 (1) \\ O(2)-C(31)-C(32) & 128 (2) & O(9)-C(39)-C(40) & 104 (1) \\ O(3)-C(33)-C(34) & 115 (2) & O(10)=C(39)-C(40) & 104 (1) \\ O(3)-C(33)-C(34) & 115 (2) & O(10)=C(39)-C(40) & 104 (1) \\ C(7)-C(7)-O(1') & 108 (1) & C(7')-C(8') & 109 (1) \\ C(7')-C(7')-C(8') & 100 (1) & O(2')-C(7')-O(8') & 120 (1) \\ C(2')-C(3')-O(4') & 110 (1) & O(2')-C(7')-C(8') & 109 (1) \\ C(2')-C(3')-O(4') & 106 (3) (9) & O(2')-C(7')-C(8') & 109 (1) \\ C(2')-C(3')-O(2') & 108 (1) & C(7')-C(8')-C(11') & 18 (1) \\ C(3')-C(4')-O(2') & 108 (1) & C(7')-C(8')-C(11') & 18 (1) \\ C(3')-C(4')-C(5') & 104 (1) & C(7')-C(8')-C(11') & 106 (1) \\ C(4')-C(5')-O(1') & 108 (1) & C(8')-C(9')-C(11') & 106 (1) \\ C(4')-C(5')-O(1') & 108 (1) & C(8')-C(9')-C(10') & 132 (1) \\ C(4')-C(5')-O(1') & 108 (1) & C(8')-C(9')-C(10') & 132 (1) \\ C(4')-C(5')-O(1') & $				
$\begin{array}{cccccccccccccccccccccccccccccccccccc$				
$\begin{array}{c} C(8)-C(9)-C(11) & 111 (1) & C(19)-C(20)-C(29) & 108 \\ C(10)-C(9)-C(11) & 112 (1) & C(19)-C(20)-C(30) & 112 \\ C(1)-C(10)-C(5) & 108 (1) & C(21)-C(20)-C(30) & 114 \\ C(1)-C(10)-C(9) & 104 (1) & C(21)-C(20)-C(30) & 114 \\ C(1)-C(10)-C(29) & 106 (1) & C(29)-C(20)-C(30) & 109 \\ C(5)-C(10)-C(9) & 106 (1) & C(20)-C(21)-C(21) & 112 \\ C(5)-C(10)-C(25) & 116 (1) & C(20)-C(21)-O(11) & 108 \\ C(9)-C(10)-C(25) & 115 (1) & C(22)-C(21)-O(11) & 106 \\ C(9)-C(11)-C(12) & 114 (1) & C(17)-C(22)-C(21) & 117 \\ C(11)-C(12)=C(13) & 125 (1) & C(17)-C(22)-O(9) & 107 \\ C(12)=C(13)-C(14) & 121 (1) & C(21)-C(22)-O(9) & 107 \\ C(12)=C(13)-C(14) & 121 (1) & C(21)-C(22)-O(9) & 105 \\ \hline \\ (ii) Extranuclear & C(24)-O(3)-C(24) & 123 (1) & O(4)=C(33)-C(34) & 131 \\ C(21)-O(11)-C(11) & 116 3 (9) & O(5)-C(35)=O(6) & 120 \\ C(28)-O(7)-C(28) & 119 (1) & O(5)-C(35)=C(36) & 111 \\ C(21)-O(11)-C(11) & 116 3 (9) & O(6)=C(35)-C(36) & 111 \\ C(21)-O(11)-C(11) & 116 3 (9) & O(6)=C(35)-C(36) & 129 \\ C(4)-C(24)-O(3) & 109 (1) & O(7)-C(37)=O(8) & 116 \\ C(7)-C(28)-O(7) & 109 (1) & O(7)-C(37)=O(8) & 116 \\ C(7)-C(28)-O(7) & 109 (1) & O(7)-C(37)=O(8) & 113 \\ O(1)-C(31)=O(2) & 125 (2) & O(8)=C(37)-C(38) & 113 \\ O(1)-C(31)=C(32) & 128 (2) & O(9)-C(39)=C(40) & 104 \\ O(3)-C(33)=C(34) & 114 (2) & & & & & & & & & & & & & & & & & & &$				
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(8)-C(9)-C(10)			104 (1)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(8)-C(9)-C(11)	111 (1)		108 (1)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(10)-C(9)-C(11)	112 (1)	C(19)-C(20)-C(30)	112 (1)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(1)-C(10)-C(5)	108 (1)	C(21)C(20)C(29)	110(1)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$				114 (1)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		107 (1)		109 (1)
$\begin{array}{c} C(5)-C(10)-C(25) & 116 (1) & C(20)-C(21)-O(11) & 108-\\ C(9)-C(11)-C(12) & 114 (1) & C(22)-C(21)-O(11) & 106-\\ C(9)-C(11)-C(12) & 114 (1) & C(17)-C(22)-C(21) & 117-\\ C(11)-C(12)=C(13) & 125 (1) & C(17)-C(22)-O(9) & 107-\\ C(12)=C(13)-C(14) & 121 (1) & C(21)-C(22)-O(9) & 105-\\ C(12)=C(13)-C(14) & 121 (1) & C(21)-C(22)-O(9) & 105-\\ C(13)-C(24) & 123 (1) & O(4)=C(33)-C(34) & 131-\\ C(16)-O(5)-C(35) & 121 (1) & O(5)-C(35)-C(36) & 120-\\ C(28)-O(7)-C(28) & 119 (1) & O(5)-C(35)-C(36) & 111-\\ C(21)-O(11)-C(1') & 116-3 (9) & O(6)=C(35)-C(36) & 129-\\ C(4)-C(24)-O(3) & 109 (1) & O(7)-C(37)-C(38) & 116-\\ C(17)-C(28)-O(7) & 109 (1) & O(7)-C(37)-C(38) & 116-\\ C(17)-C(28)-O(7) & 109 (1) & O(7)-C(37)-C(38) & 113-\\ O(1)-C(31)-C(32) & 106 (2) & O(9)-C(39)-C(40) & 104-\\ O(3)-C(33)-C(32) & 128 (2) & O(9)-C(39)-C(40) & 104-\\ O(3)-C(33)-C(34) & 114 (2) & & & & & & & & & & & & & & & & & & &$		106 (1)		112 (1)
$\begin{array}{c} C(9)-C(10)-C(25) & 115 (1) & C(22)-C(21)-O(11) & 106-\\ C(9)-C(11)-C(12) & 114 (1) & C(17)-C(22)-C(21) & 117-\\ C(11)-C(12)=C(13) & 125 (1) & C(17)-C(22)-O(9) & 107-\\ C(12)=C(13)-C(14) & 121 (1) & C(21)-C(22)-O(9) & 105-\\ (ii) Extranuclear & & & & & & & & \\ C(24)-O(3)-C(24) & 123 (1) & O(4)=C(33)-C(34) & 131-\\ C(16)-O(5)-C(35) & 121 (1) & O(5)-C(35)-O(6) & 120-\\ C(28)-O(7)-C(28) & 119 (1) & O(5)-C(35)-C(36) & 111-\\ C(21)-O(11)-C(1') & 116-3 (9) & O(6)=C(35)-C(36) & 129-\\ C(4)-C(24)-O(3) & 109 (1) & O(7)-C(37)-C(38) & 113-\\ O(1)-C(31)-O(2) & 125 (2) & O(8)=C(37)-C(38) & 129-\\ O(1)-C(31)-C(32) & 106 (2) & O(9)-C(39)-O(10) & 125-\\ O(2)=C(31)-C(32) & 128 (2) & O(9)-C(39)-C(40) & 104-\\ O(3)-C(33)-C(34) & 115 (2) & O(10)=C(39)-C(40) & 130-\\ O(3)-C(33)-C(34) & 114 (2) & & & & & \\ (iii) Glucopyranosyl moiety & & & & & \\ O(11)-C(1')-C(2') & 110 (1) & C(2')-O(4')-C(12') & 117-\\ O(11)-C(1')-O(1') & 108-8-(9) & O(2')-C(7')-O(8') & 109-\\ C(1')-C(2')-C(3') & 110 (1) & O(2'')-C(7'')-C(8'') & 109-\\ C(1')-C(2')-C(3') & 104 (1) & O(2'')-C(7'')-C(8'') & 126-\\ C(3')-C(2')-C(4') & 110 (1) & O(2'')-C(7'')-C(8'') & 124-\\ C(2')-C(3')-O(2') & 108 (1) & C(7')-C(8')-C(11') & 118-\\ C(3')-C(4')-O(2') & 108 (1) & C(7')-C(8')-C(11') & 118-\\ C(3')-C(4')-O(2') & 108 (1) & C(7')-C(8')-C(11') & 118-\\ C(3')-C(4')-O(2') & 108 (1) & C(7')-C(8')-C(11') & 130-\\ C(4')-C(5')-O(1') & 108 (1) & C(8'')-C(8'')-C(11'') & 125-\\ C(4')-C(5')-O(1') & 108 (1) & C(8'')-C(8'')-C(11'') & 125-\\ C(4')-C(5')-O(1') & 108 (1) & C(8'')-C(9'')-C(11'') & 125-\\ C(4')-C(5')-O(1') & 108 (1) & C(8'')-C(9'')-C(10'') & 125-\\ C(4')-C(5')-O(1') & 108 (1) & C(8'')-C(12')-C(10'') & 125-\\ C(4')-C(5')-O(1') & 108 (1) & C(8'')-C(12')-C(10'') & 125-\\ C(4')-C(5')-O(1') & 108 (1) & C(8'')-C(12')-C(10'') & 125-\\ C(4')-C(5')-O(1') & 108 (1) & C(8'')-$			C(20) C(21) O(11)	108-5 (9)
$\begin{array}{c} C(9)-C(11)-C(12) & 114 (1) & C(17)-C(22)-C(21) & 117 \cdot C(11)-C(12)-C(13) & 125 (1) & C(17)-C(22)-O(9) & 107 \cdot C(12)-C(13)-C(14) & 121 (1) & C(21)-C(22)-O(9) & 105 \cdot C(12)-C(12)-C(14) & 121 (1) & C(21)-C(22)-O(9) & 105 \cdot C(12)-C(12)-C(14) & 121 (1) & C(21)-C(22)-O(9) & 105 \cdot C(12)-C(12)-C(14) & 121 (1) & C(21)-C(22)-O(9) & 105 \cdot C(12)-C(12)-C(12) & 110 \cdot C(11) & O(5)-C(35)-C(34) & 131 \cdot C(16)-O(5)-C(35) & 121 (1) & O(5)-C(35)-O(6) & 120 \cdot C(28)-O(7)-C(28) & 119 (1) & O(5)-C(35)-C(36) & 129 \cdot C(21)-O(11)-C(1') & 116 \cdot 3 (9) & O(6)-C(35)-C(36) & 129 \cdot C(21)-O(11)-C(1') & 116 \cdot 3 (9) & O(6)-C(35)-C(36) & 129 \cdot C(21)-C(24)-O(3) & 109 (1) & O(7)-C(37)-C(38) & 116 \cdot C(17)-C(28)-O(7) & 109 (1) & O(7)-C(37)-C(38) & 113 \cdot O(1)-C(31)-C(32) & 106 \cdot (2) & O(9)-C(39)-C(10) & 125 \cdot (2) & O(8)-C(37)-C(38) & 129 \cdot O(2)-C(31)-C(32) & 128 \cdot (2) & O(9)-C(39)-C(40) & 104 \cdot O(3)-C(33)-C(33)-O(34) & 114 \cdot (2) & O(10)-C(39)-C(40) & 130 \cdot O(3)-C(33)-C(34) & 114 \cdot (2) & O(10)-C(39)-C(40) & 130 \cdot O(3)-C(33)-C(34) & 114 \cdot (2) & O(10)-C(7)-C(8') & 130 \cdot O(11)-C(1')-O(1') & 108 \cdot 8 \cdot (9) & O(2')-C(7')-O(8') & 130 \cdot O(11)-C(1')-O(1') & 108 \cdot 8 \cdot (9) & O(2')-C(7')-O(8') & 130 \cdot O(11)-C(1')-O(1') & 106 \cdot 3 \cdot (9) & O(2'')-C(7')-O(8') & 130 \cdot O(1)-C(1')-C(2')-C(4') & 110 \cdot (1) & O(2'')-C(7')-O(8') & 132 \cdot C(1')-C(2')-C(4') & 110 \cdot (1) & O(3'')-C(7')-C(8'') & 124 \cdot C(2')-C(3')-O(4') & 110 \cdot (1) & O(3'')-C(7')-C(8'') & 124 \cdot C(2')-C(3')-O(2') & 108 \cdot (1) & C(7')-C(8')-C(11') & 130 \cdot C(3')-C(4')-O(2') & 108 \cdot (1) & C(7')-C(8')-C(11') & 130 \cdot C(3')-C(4')-O(2') & 108 \cdot (1) & C(7')-C(8')-C(11') & 130 \cdot C(3')-C(4')-O(2') & 108 \cdot (1) & C(7')-C(8')-C(11') & 130 \cdot C(3')-C(4')-O(2') & 108 \cdot (1) & C(7')-C(8')-C(11') & 125 \cdot C(4')-C(5')-O(1') & 108 \cdot (1) & C(8')-C(9')-C(11') & 125 \cdot C(4')-C(5')-O(1') & 108 \cdot (1) & C(8')-C(9')-C(11') & 125 \cdot C(4')-C(5')-O(1') & 108 \cdot (1) & C(8')-C(9'')-C(10'') & 134 \cdot C(4')-C(5')-O(1') & 108 \cdot (1) & C(8')-C(9'')-C(10'') & 134 \cdot C(4')-C(5')-O(1') & 108 \cdot (1) & C(8')-C(9'')-C(10'') & 132 \cdot C(1')-O(1')-C(1') & 108 \cdot (1) &$				
$\begin{array}{c} C(11)-C(12)=C(13) & 125 (1) & C(17)-C(22)-O(9) & 107 \cdot C(12)=C(13)-C(14) & 121 (1) & C(21)-C(22)-O(9) & 105 \cdot C(21)-C(21)-C(24) & 123 (1) & O(4)=C(33)-C(34) & 131 \cdot C(16)-O(5)-C(35) & 121 (1) & O(5)-C(35)-O(6) & 120 \cdot C(28)-O(7)-C(28) & 119 (1) & O(5)-C(35)-C(36) & 111 \cdot C(21)-O(11)-C(1') & 116 \cdot 3 (9) & O(6)=C(35)-C(36) & 111 \cdot C(21)-O(11)-C(1') & 116 \cdot 3 (9) & O(6)=C(35)-C(36) & 111 \cdot C(21)-O(11)-C(1') & 116 \cdot 3 (9) & O(6)=C(35)-C(36) & 116 \cdot C(17)-C(28)-O(7) & 109 (1) & O(7)-C(37)-C(38) & 116 \cdot C(17)-C(28)-O(7) & 109 (1) & O(7)-C(37)-C(38) & 113 \cdot O(1)-C(31)-C(32) & 106 \cdot (2) & O(9)-C(39)=O(10) & 125 \cdot C(10)-C(31)-C(32) & 106 \cdot (2) & O(9)-C(39)=O(10) & 125 \cdot C(10)-C(31)-C(32) & 106 \cdot (2) & O(9)-C(39)-C(40) & 104 \cdot C(17)-C(17)-C(17) & 114 \cdot (2) & O(10)-C(17)-C(27) & 110 \cdot (1) & C(27)-C(40) & 130 \cdot C(33)-C(33)-C(34) & 114 \cdot (2) & O(10)-C(39)-C(40) & 130 \cdot C(37)-C(17)-O(17) & 108 \cdot 8 (9) & O(27)-C(77)-O(37) & 119 \cdot C(27)-C(17)-O(17) & 108 \cdot 8 (9) & O(27)-C(77)-O(87) & 109 \cdot C(17)-C(27)-C(47) & 110 \cdot (1) & O(37)-C(77)-C(87) & 132 \cdot C(17)-C(27)-C(47) & 110 \cdot (1) & O(37)-C(77)-C(87) & 132 \cdot C(17)-C(27)-C(47) & 110 \cdot (1) & O(37)-C(77)-C(87) & 124 \cdot C(27)-C(37)-O(27) & 104 \cdot (1) & C(77)-C(87)-C(87) & 124 \cdot C(27)-C(37)-O(27) & 108 \cdot (1) & C(77)-C(87)-C(117) & 18 \cdot C(27)-C(47)-O(27) & 108 \cdot (1) & C(77)-C(87)-C(117) & 130 \cdot C(37)-C(47)-O(27) & 108 \cdot (1) & C(77)-C(87)-C(117) & 125 \cdot C(47)-C(57) & 104 \cdot (1) & C(77)-C(87)-C(117) & 125 \cdot C(47)-C(57)-O(17) & 108 \cdot (1) & C(87)-C(87)-C(117) & 125 \cdot C(47)-C(57)-O(17) & 108 \cdot (1) & C(87)-C(87)-C(117) & 125 \cdot C(47)-C(57)-O(17) & 108 \cdot (1) & C(87)-C(87)-C(117) & 125 \cdot C(47)-C(57)-O(17) & 108 \cdot (1) & C(87)-C(87)-C(117) & 125 \cdot C(47)-C(57)-O(17) & 108 \cdot (1) & C(87)-C(97)-C(107) & 132 \cdot C(47)-O(27)-O(17) & 108 \cdot (1) & C(87)-C(97)-C(107) & 132 \cdot C(47)-O(27)-O(17) & 108 \cdot (1) & C(87)-C(97)-C(107) & 132 \cdot C(47)-O(27)-O(17) & 108 \cdot (1) & C(87)-C(97)-C(107) & 132 \cdot C(47)-O(27)-O(17) & 108 \cdot (1) & C(87)-C(97)-C(107) & 132 \cdot C(47)-O(27)-O(17) & 108 \cdot (1) & C(87)-C(97)-C(107) & 132 \cdot$				106-4 (9)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$			C(17) - C(22) - C(21)	117 (1)
(ii) Extranuclear C(24)—0(3)—C(24) 123 (1) 0(4)=C(33)—C(34) 131 (1) C(16)—0(5)—C(35) 121 (1) 0(5)—C(35)=O(6) 120 (1) (28)—0(7)—C(28) 119 (1) 0(5)—C(35)—C(36) 129 (1) (21)—0(11)—C(1') 116·3 (9) 0(6)=C(35)—C(36) 129 (1) (21)—0(11)—C(1') 116·3 (9) 0(6)=C(35)—C(36) 129 (1) (21)—0(13)—O(2) 125 (2) 0(8)=C(37)—C(38) 113 (1) (1)—C(31)—O(2) 125 (2) 0(8)=C(37)—C(38) 129 (1) (1)—C(31)—C(32) 106 (2) 0(9)—C(39)=O(10) 125 (2) 0(3)—C(33)=O(10) 125 (2) 0(9)—C(39)=O(10) 125 (2) 0(3)—C(33)=O(4) 115 (2) 0(10)=C(39)—C(40) 104 (1) (3)—C(33)—C(34) 114 (2) (10)—C(1')—C(2') 110 (1) C(2')—O(4')—C(12') 117 (1) (1)—C(1')—O(1') 108·8 (9) 0(2')—C(7')—O(8') 109 (1)—C(1')—C(2')—10 (1) 0(3)—C(7')—C(8') 109 (1)—C(1')—C(2')—C(3') 110 (1) 0(3')—C(7')—C(8') 132 (1')—C(2')—C(4') 110 (1) 0(3')—C(7')—C(8') 132 (1')—C(2')—C(4') 110 (1) 0(3')—C(7')—C(8') 126 (1)—C(2')—C(3')—O(4') 110 (1) 0(3')—C(7')—C(8') 126 (1)—C(2')—C(3')—O(2') 108 (1) C(7')—C(8')=C(9') 112 (1) C(2')—C(3')—O(2') 108 (1) C(7')—C(8')=C(1') 118 (1) C(3')—C(4')—O(2') 108 (1) C(7')—C(8')=C(1') 118 (1) C(3')—C(4')—O(2') 108 (1) C(7')—C(8')=C(1') 129 (1) C(3')—C(1')—O(1') 108 (1) C(8')=C(9')—C(11') 125 (1) C(4')—C(5')—O(1') 108 (1) C(8')=C(9')—C(10') 134 (1) C(1')—O(1')—C(1') 116 (1) C(1')—O(1') 116 (1) C(1')—O(1') 108 (1) C(8')=C(9')—C(10') 134 (1) C(1')—O(1')—O(1') 108 (1) C(1')—O(1')—O(1') 109 (1) O(1)—O(1')—O(1') 109 (1) O(1)—O(1')—O(1') 100 (1) O(1)—O(1')—O(1') 100	C(11)-C(12)=C(13)			107-2 (9)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(12)=C(13)-C(14)	121 (1)	C(21)-C(22)-O(9)	105-4 (9)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$				
$\begin{array}{c} C(16)-O(5)-C(35) & 121 (1) & O(5)-C(35)=O(6) & 120 (1) \\ C(28)-O(7)-C(28) & 119 (1) & O(5)-C(35)-C(36) & 111 (1) \\ C(21)-O(11)-C(1') & 116-3 (9) & O(6)=C(35)-C(36) & 129 (1) \\ C(4)-C(24)-O(3) & 109 (1) & O(7)-C(37)=O(8) & 116 (1) \\ C(17)-C(28)-O(7) & 109 (1) & O(7)-C(37)-C(38) & 129 (1) \\ O(1)-C(31)+O(2) & 125 (2) & O(8)=C(37)-C(38) & 129 (1) \\ O(1)-C(31)-C(32) & 106 (2) & O(9)-C(39)=O(10) & 125 (1) \\ O(2)=C(31)+C(32) & 128 (2) & O(9)-C(39)+C(40) & 104 (1) \\ O(3)-C(33)=O(4) & 115 (2) & O(10)=C(39)-C(40) & 130 (1) \\ O(3)-C(33)+O(4) & 115 (2) & O(10)=C(39)-C(40) & 130 (1) \\ O(3)-C(33)+O(4) & 114 (2) & & & & & \\ \hline \\ (iii) Glucopyranosyl moiety & & & & & \\ O(11)-C(1')-O(1') & 108-8 (9) & O(2')-C(7')-O(3') & 119 (1) \\ C(2')-C(1')-O(1') & 109 (1) & O(2')-C(7')-C(8') & 133 (1) \\ C(1')-C(2')-C(3') & 110 (1) & O(2')-C(7')-O(8') & 132 (1) \\ C(3')-C(2')-O(4') & 110 (1) & O(2'')-C(7'')-C(8'') & 126 (1) \\ C(2')-C(3')-O(4') & 110 (1) & O(2'')-C(7'')-C(8'') & 124 (1) \\ C(2')-C(3')-O(2'') & 104 (1) & C(7')-C(8')=C(9') & 112 (1) \\ C(3')-C(4')-C(5') & 112 (1) & C(9')+C(8')+C(11') & 130 (1) \\ C(3')-C(4')-C(5') & 108 (1) & C(7')-C(8')+C(11') & 130 (1) \\ C(3')-C(4')-C(5') & 104 (1) & C(7')-C(8')-C(11') & 130 (1) \\ C(3')-C(4')-C(5') & 104 (1) & C(7')-C(8')-C(11') & 130 (1) \\ C(3')-C(4')-C(5') & 104 (1) & C(7')-C(8')+C(11') & 130 (1) \\ C(8')-C(5')-O(1') & 108 (1) & C(8')+C(9')-C(10'') & 124 (1) \\ C(8')-C(5')-O(1') & 108 (1) & C(8')+C(9')-C(10'') & 134 (1) \\ C(1')-O(1')-C(1') & 108 (1) & C(8')+C(9')-C(10'') & 132 (1) \\ C(1')-O(1')-C(1') & 108 (1) & C(8')+C(12')-C(10'') & 132 (1) \\ C(1')-O(1')-C(1') & 108 (1) & C(8')+C(12')-C(10'') & 132 (1) \\ C(1')-O(1')-C(1') & 101 (1) & O(4')-C(12')+O(1'') & 125 (1) \\ C(4')-C(5')-O(1') & 108 (1) & C(8')+C(12')-C(10'') & 132 (1) \\ C(4')-C(5')-C(7') & 120 (1) & O(4')-C(12')-C(13'') & 112 (1) \\ C(4')-C(5')-C(7') & 120 (1) & O(4')-C(12')-C(13'') & 112 (1) \\ C(4')-C(5')-C(7') & 120 (1) & O(4')-C(12')-C(13'') & 112 (1) \\ C(4')-C(12')-C(1'') & 100 (1) & O(4')-C(12')-C(13'') & 112 (1) \\ C(4')-C(12')-$	(ii) Extranuclear			
$\begin{array}{c} C(16)-O(5)-C(35) & 121 (1) & O(5)-C(35)=O(6) & 120 (1) \\ C(28)-O(7)-C(28) & 119 (1) & O(5)-C(35)-C(36) & 111 (1) \\ C(21)-O(11)-C(1') & 116-3 (9) & O(6)=C(35)-C(36) & 129 (1) \\ C(4)-C(24)-O(3) & 109 (1) & O(7)-C(37)=O(8) & 116 (1) \\ C(17)-C(28)-O(7) & 109 (1) & O(7)-C(37)-C(38) & 129 (1) \\ O(1)-C(31)+O(2) & 125 (2) & O(8)=C(37)-C(38) & 129 (1) \\ O(1)-C(31)-C(32) & 106 (2) & O(9)-C(39)=O(10) & 125 (1) \\ O(2)=C(31)+C(32) & 128 (2) & O(9)-C(39)+C(40) & 104 (1) \\ O(3)-C(33)=O(4) & 115 (2) & O(10)=C(39)-C(40) & 130 (1) \\ O(3)-C(33)+O(4) & 115 (2) & O(10)=C(39)-C(40) & 130 (1) \\ O(3)-C(33)+O(4) & 114 (2) & & & & & \\ \hline \\ (iii) Glucopyranosyl moiety & & & & & \\ O(11)-C(1')-O(1') & 108-8 (9) & O(2')-C(7')-O(3') & 119 (1) \\ C(2')-C(1')-O(1') & 109 (1) & O(2')-C(7')-C(8') & 133 (1) \\ C(1')-C(2')-C(3') & 110 (1) & O(2')-C(7')-O(8') & 132 (1) \\ C(3')-C(2')-O(4') & 110 (1) & O(2'')-C(7'')-C(8'') & 126 (1) \\ C(2')-C(3')-O(4') & 110 (1) & O(2'')-C(7'')-C(8'') & 124 (1) \\ C(2')-C(3')-O(2'') & 104 (1) & C(7')-C(8')=C(9') & 112 (1) \\ C(3')-C(4')-C(5') & 112 (1) & C(9')+C(8')+C(11') & 130 (1) \\ C(3')-C(4')-C(5') & 108 (1) & C(7')-C(8')+C(11') & 130 (1) \\ C(3')-C(4')-C(5') & 104 (1) & C(7')-C(8')-C(11') & 130 (1) \\ C(3')-C(4')-C(5') & 104 (1) & C(7')-C(8')-C(11') & 130 (1) \\ C(3')-C(4')-C(5') & 104 (1) & C(7')-C(8')+C(11') & 130 (1) \\ C(8')-C(5')-O(1') & 108 (1) & C(8')+C(9')-C(10'') & 124 (1) \\ C(8')-C(5')-O(1') & 108 (1) & C(8')+C(9')-C(10'') & 134 (1) \\ C(1')-O(1')-C(1') & 108 (1) & C(8')+C(9')-C(10'') & 132 (1) \\ C(1')-O(1')-C(1') & 108 (1) & C(8')+C(12')-C(10'') & 132 (1) \\ C(1')-O(1')-C(1') & 108 (1) & C(8')+C(12')-C(10'') & 132 (1) \\ C(1')-O(1')-C(1') & 101 (1) & O(4')-C(12')+O(1'') & 125 (1) \\ C(4')-C(5')-O(1') & 108 (1) & C(8')+C(12')-C(10'') & 132 (1) \\ C(4')-C(5')-C(7') & 120 (1) & O(4')-C(12')-C(13'') & 112 (1) \\ C(4')-C(5')-C(7') & 120 (1) & O(4')-C(12')-C(13'') & 112 (1) \\ C(4')-C(5')-C(7') & 120 (1) & O(4')-C(12')-C(13'') & 112 (1) \\ C(4')-C(12')-C(1'') & 100 (1) & O(4')-C(12')-C(13'') & 112 (1) \\ C(4')-C(12')-$	C(24)-O(3)-C(24)	123 (1)	O(4)=C(33)-C(34)	131 (2)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(16)=O(5)=C(35)		O(5)-C(35)=O(6)	120(1)
$\begin{array}{c} C(2) - O(11) - C(1') & 116 \cdot 3 \cdot (9) & O(6) = C(35) - C(36) & 129 \cdot (1) - C(34) - O(3) & 109 \cdot (1) & O(7) - C(37) - O(8) & 116 \cdot (1) - C(28) - O(7) & 109 \cdot (1) & O(7) - C(37) - C(38) & 113 \cdot (1) - C(31) = O(2) & 125 \cdot (2) & O(8) = C(37) - C(38) & 113 \cdot (1) - C(31) = C(32) & 125 \cdot (2) & O(9) - C(39) - C(108) & 129 \cdot (1) - C(31) - C(32) & 128 \cdot (2) & O(9) - C(39) - C(40) & 104 \cdot (1) - C(33) - C(32) & 128 \cdot (2) & O(9) - C(39) - C(40) & 130 \cdot (1) - C(33) - C(34) & 115 \cdot (2) & O(10) = C(39) - C(40) & 130 \cdot (1) - C(1') - C(2') & 110 \cdot (1) & C(2') - C(1') - C(2') & 110 \cdot (1) & C(2') - C(1') - O(1') & 108 \cdot 8 \cdot (9) & O(2') - C(7') - O(8') & 119 \cdot (1) - C(1') - C(1') & 109 \cdot (1) & O(2') - C(7') - C(8') & 109 \cdot (1') - C(2') - C(3') & 110 \cdot (1) & O(2'') - C(7'') - C(8'') & 109 \cdot (1') - C(2') - C(3') & 110 \cdot (1) & O(2'') - C(7'') - C(8'') & 112 \cdot (1') - C(2') - C(4') & 110 \cdot (1) & O(3'') - C(7'') - C(8'') & 112 \cdot (1') - C(2') - C(3') - O(2'') & 104 \cdot (1) & C(7') - C(8') - C(11') & 118 \cdot (1') - C(8') - C(11') & 118 \cdot (1') - C(8') - C(11') & 118 \cdot (1') - C(8'') - C(1') & 108 \cdot (1) & C(7') - C(8'') - C(11') & 118 \cdot (1') - C(8') - C(1') & 108 \cdot (1) & C(7'') - C(8'') - C(11') & 118 \cdot (1'') - C(8'') - C(1'') & 108 \cdot (1) & C(7'') - C(8'') - C(11'') & 125 \cdot (1'') - C(5') - O(1') & 108 \cdot (1) & C(8'') - C(8'') - C(11'') & 125 \cdot (1'') - C(5') - O(1') & 108 \cdot (1) & C(8'') - C(9'') - C(11'') & 125 \cdot (1'') - C(5') - O(1') & 108 \cdot (1) & C(8'') - C(9'') - C(10'') & 132 \cdot (1'') - O(1'') - O(1'') & 108 \cdot (1) & C(8'') - C(9'') - C(10'') & 132 \cdot (1'') - O(1'') - O(1'') & 108 \cdot (1) & C(8'') - C(9'') - C(10'') & 132 \cdot (1'') - O(1'') - O(1'') & 108 \cdot (1) & C(8'') - C(9'') - C(10'') & 132 \cdot (1'') - O(1'') - O(1'') & 108 \cdot (1) & C(8'') - C(9'') - C(10'') & 132 \cdot (1'') - O(1'') - O(1'') & 108 \cdot (1) & C(8'') - C(9'') - C(10'') & 132 \cdot (1'') - O(1'') - O(1'') & 108 \cdot (1) & C(8'') - C(9'') - C(10'') & 132 \cdot (1'') - O(1'') - O(1'') & 108 \cdot (1) & C(8'') - C(9'') - C(10'') & 132 \cdot (1'') - O(1'') - O(1'') & 108 \cdot (1) & C(8'') - C(10'') - C(10'') & $				111 (1)
$\begin{array}{c} C(4)-C(24)-O(3) & 109 \ (1) & O(7)-C(37)=O(8) & 116 \ C(17)-C(28)-O(7) & 109 \ (1) & O(7)-C(37)=O(8) & 113 \ O(1)-C(31)=O(2) & 125 \ (2) & O(8)=C(37)-C(38) & 129 \ O(1)-C(31)-C(32) & 106 \ (2) & O(9)-C(39)=O(10) & 125 \ O(3)-C(33)=O(4) & 115 \ (2) & O(10)=C(39)-C(40) & 104 \ O(3)-C(33)-C(34) & 114 \ (2) & & & & & & & & & & & & & & & & & & &$				
$\begin{array}{cccccccccccccccccccccccccccccccccccc$				116 (2)
$\begin{array}{c} O(1)-C(31)=O(2) & 125 (2) & O(8)=C(37)-C(38) & 129 (2) \\ O(1)-C(31)-C(32) & 106 (2) & O(9)-C(39)=O(10) & 125 (2) (2) (2) (2) (2) (2) (2) (2) (2) (2)$				
$\begin{array}{cccccccccccccccccccccccccccccccccccc$				
$\begin{array}{cccccccccccccccccccccccccccccccccccc$				129 (2)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	O(1)-C(31)-C(32)	106 (2)	O(9)-C(39)=O(10)	125 (2)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	O(2)=C(31)=C(32)	128 (2)	O(9)C(39)C(40)	104 (1)
$\begin{array}{llll} &0(3)-C(33)-C(34)&114\ (2)\\ &(iii)\ Glucopyranosyl moiety\\ &O(11)-C(1')-C(2')&110\ (1)&C(2')-O(4')-C(12')&117\ (0(11)-C(1')-O(1')&108.8\ (9)&O(2')-C(7')-O(3')&119\ (C(2')-C(1')-O(1')&109\ (1)&O(2')-C(7')-C(8')&109\ (C(1')-C(2')-C(3')&110\ (1)&O(2')-C(7')-C(8')&132\ (C(1')-C(2')-C(4')&110\ (1)&O(2'')-C(7'')-O(8'')&126\ (C(2')-C(3')-O(4')&110\ (1)&O(3'')-C(7'')-C(8'')&110\ (C(2')-C(3')-O(2')&104\ (1)&C(7')-C(8')-C(9'')&112\ (C(2')-C(3')-O(2')&108\ (1)&C(7')-C(8')-C(11')&130\ (C(3')-C(4')-C(5')&112\ (1)&C(9')-C(8')-C(11')&130\ (C(3')-C(4')-C(5')&112\ (1)&C(9')-C(8'')-C(11')&130\ (C(3')-C(4')-C(5')&112\ (1)&C(9')-C(8'')-C(11')&130\ (C(3')-C(4')-C(5')&113\ (1)&C(9'')-C(8'')-C(11'')&108\ (1)&C(7'')-C(8'')-C(11'')&108\ (1)&C(7'')-C(8'')-C(11'')&134\ (C(8')-C(5')-O(1')&108\ (1)&C(8'')-C(9'')-C(10'')&134\ (C(8')-C(5')-O(1')&108\ (1)&C(8'')-C(9'')-C(10'')&132\ (C(1')-O(1')-C(1')&111-6\ (9)&O(4')-C(12')-C(5')&125\ (C(4')-C(12')-C(1'')&110\ (1)&O(4')-C(12')-C(13')&1125\ (C(4')-O(2')-C(7')&120\ (1)&O(4')-C(12')-C(13')&1125\ (C(4')-O(2')-C(7')&120\ (1)&O(4')-C(12')-C(13')&1125\ (C(4')-O(2')-C(7')&120\ (1)&O(4')-C(12')-C(13')&1125\ ($		115 (2)	O(10)=C(39)-C(40)	130 (2)
(iii) Glucopyranosyl moiety $O(11)-C(1')-C(2') = 110 (1)$ $C(2')-O(4')-C(12') = 117 (1)$ $O(11)-C(1')-O(1') = 108 \cdot 8 (9) = O(2')-C(7')-O(3') = 119 (1)$ $C(2')-C(1')-O(1') = 109 \cdot 10 (1) = 0(2')-C(7')-C(8') = 109 \cdot 10 (1) = 0(2')-C(3') = 110 (1) = 0(3')-C(7')-C(8') = 132 (1')-C(2')-C(4') = 110 (1) = O(2'')-C(7'')-O(8'') = 126 (1')-C(2')-C(3')-O(4') = 110 (1) = O(3'')-C(7'')-C(8'') = 124 (1) (1) = 0(2')-C(3')-O(2'') = 104 (1) = 0(3'')-C(7')-C(8'') = 124 (1) (1) = 0(3'')-C(3')-O(2'') = 108 (1) = C(7')-C(8')-C(11') = 118 (1) = 0(3')-C(4')-O(2') = 108 (1) = C(7')-C(8'')-C(11') = 118 (1) = 0(3')-C(4')-O(2') = 108 (1) = C(7')-C(8'')-C(11') = 130 (1) = 0(2')-C(4')-C(5') = 104 (1) = C(7'')-C(8'')-C(11') = 106 (1) = 0(4')-C(5')-O(1') = 108 (1) = C(8'')-C(1'')-C(11'') = 125 (1) = 0(2')-C(1')-O(1') = 108 (1) = C(8'')-C(10'') = 134 (1) = 0(4')-C(5')-O(1') = 108 (1) = C(8'')-C(10'') = 132 (1) = 0(4')-C(12')-C(1'') = 108 (1) = C(8'')-C(10'') = 132 (1) = 0(4')-C(12')-C(1'') = 105 (1) = 0(4')-C(12')-C(10'') = 125 (1) = 0(4')-C(12')-C(1'') = 112 (1) = 0(4')-C(12')-C(13'') = 0(4')-C(12')-C(13'') = 112 (1) = 0(4')-C(12')-C(13'') = 0(4')-C(12')-C($, , , , ,	-
$\begin{array}{llllllllllllllllllllllllllllllllllll$	- (-) - (-)	• •		
$\begin{array}{llllllllllllllllllllllllllllllllllll$	(iii) Glucopyranosyl moie	tv		
$\begin{array}{llllllllllllllllllllllllllllllllllll$	0(1)-((1)-((2)	110(1)	C(2')O(4')C(12')	117(1)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$				119 (2)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$				
$\begin{array}{llll} C(1')-C(2')-C(4') & 110 \ (1) & O(2'')-C(7'')-O(3'') & 1264 \ C(3')-C(2')-O(4') & 106\cdot3 \ (9) & O(2'')-C(7'')-C(8'') & 1104 \ C(2')-C(3')-C(4') & 110 \ (1) & O(3'')-C(7'')-C(8'') & 1244 \ C(2')-C(3')-O(2'') & 104 \ (1) & C(7')-C(8')-C(9') & 1124 \ C(3')-C(4')-C(5') & 112 \ (1) & C(9')-C(8')-C(11') & 1364 \ C(3')-C(4')-C(5') & 112 \ (1) & C(9')-C(8'')-C(11') & 1364 \ C(3')-C(4')-C(5') & 104 \ (1) & C(7'')-C(8'')-C(11') & 1064 \ C(4')-C(5')-C(6') & 113 \ (1) & C(9'')-C(8'')-C(11'') & 1264 \ C(4')-C(5')-O(1') & 108 \ (1) & C(8'')-C(9'')-C(10'') & 1344 \ C(8'')-C(5')-O(1') & 108 \ (1) & C(8'')-C(9'')-C(10'') & 1324 \ C(1')-O(1')-C(1') & 111\cdot6 \ (9) & O(4')-C(12')-C(10'') & 1254 \ C(4')-O(2')-C(7') & 120 \ (1) & O(4')-C(12')-C(13') & 1125 \ \end{array}$	C(2)-C(1)-O(1)			
$\begin{array}{lllll} & C(2')-C(3')-C(4') & 110 \ (1) & O(3'')-C(7'')-C(8'') & 124 \ (C(2')-C(3')-O(2'') & 104 \ (1) & C(7')-C(8')-C(8'') & 112 \ (C(3')-C(3')-O(2') & 108 \ (1) & C(7')-C(8')-C(11') & 118 \ (C(3')-C(4')-O(2') & 108 \ (1) & C(7'')-C(8'')-C(9'') & 129 \ (O(2')-C(4')-O(2') & 104 \ (1) & C(7'')-C(8'')-C(9'') & 129 \ (C(4')-C(5')-C(6') & 113 \ (1) & C(9'')-C(8'')-C(11'') & 125 \ (C(4')-C(5')-O(1') & 108 \ (1) & C(8'')-C(9')-C(10'') & 134 \ (C(8')-C(5')-O(1') & 108 \ (1) & C(8'')-C(9'')-C(10'') & 132 \ (C(1')-O(1')-C(1') & 111 \cdot 6 \ (9) & O(4')-C(12')-O(5') & 125 \ (C(4')-O(2')-C(7') & 120 \ (1) & O(4')-C(12')-C(13') & 1125 \ \end{array}$	C(1')-C(2')-C(3')		0(3')=C(7')=C(8')	
$\begin{array}{lllll} & C(2')-C(3')-C(4') & 110 \ (1) & O(3'')-C(7'')-C(8'') & 124 \ (C(2')-C(3')-O(2'') & 104 \ (1) & C(7')-C(8')-C(8'') & 112 \ (C(3')-C(3')-O(2') & 108 \ (1) & C(7')-C(8')-C(11') & 118 \ (C(3')-C(4')-O(2') & 108 \ (1) & C(7'')-C(8'')-C(9'') & 129 \ (O(2')-C(4')-O(2') & 104 \ (1) & C(7'')-C(8'')-C(9'') & 129 \ (C(4')-C(5')-C(6') & 113 \ (1) & C(9'')-C(8'')-C(11'') & 125 \ (C(4')-C(5')-O(1') & 108 \ (1) & C(8'')-C(9')-C(10'') & 134 \ (C(8')-C(5')-O(1') & 108 \ (1) & C(8'')-C(9'')-C(10'') & 132 \ (C(1')-O(1')-C(1') & 111 \cdot 6 \ (9) & O(4')-C(12')-O(5') & 125 \ (C(4')-O(2')-C(7') & 120 \ (1) & O(4')-C(12')-C(13') & 1125 \ \end{array}$			O(2")C(7")O(3")	126 (2)
$\begin{array}{lllll} & C(2')-C(3')-C(4') & 110 \ (1) & O(3'')-C(7'')-C(8'') & 124 \ (C(2')-C(3')-O(2'') & 104 \ (1) & C(7')-C(8')-C(8'') & 112 \ (C(3')-C(3')-O(2') & 108 \ (1) & C(7')-C(8')-C(11') & 118 \ (C(3')-C(4')-O(2') & 108 \ (1) & C(7'')-C(8'')-C(9'') & 129 \ (O(2')-C(4')-O(2') & 104 \ (1) & C(7'')-C(8'')-C(9'') & 129 \ (C(4')-C(5')-C(6') & 113 \ (1) & C(9'')-C(8'')-C(11'') & 125 \ (C(4')-C(5')-O(1') & 108 \ (1) & C(8'')-C(9')-C(10'') & 134 \ (C(8')-C(5')-O(1') & 108 \ (1) & C(8'')-C(9'')-C(10'') & 132 \ (C(1')-O(1')-C(1') & 111 \cdot 6 \ (9) & O(4')-C(12')-O(5') & 125 \ (C(4')-O(2')-C(7') & 120 \ (1) & O(4')-C(12')-C(13') & 1125 \ \end{array}$	C(3')-C(2')-O(4')	106-3 (9)	O(2")-C(7")-C(8")	110(1)
$\begin{array}{llll} C(2')-C(3')-O(2'') & 104 & (1) & C(7')-C(8')=C(9') & 112 \\ C(4')-C(3')-O(2') & 108 & (1) & C(7')-C(8')-C(11') & 118 \\ C(3')-C(4')-C(5') & 112 & (1) & C(9')=C(8')-C(11') & 130 \\ C(3')-C(4')-C(5') & 108 & (1) & C(7'')-C(8'')-C(9'') & 129 \\ O(2')-C(4')-C(5') & 104 & (1) & C(7'')-C(8'')-C(11'') & 106 \\ C(4')-C(5')-C(6') & 113 & (1) & C(9'')-C(8'')-C(11'') & 125 \\ C(4')-C(5')-O(1') & 108 & (1) & C(8')=C(9')-C(10'') & 134 \\ C(8')-C(5')-O(1') & 108 & (1) & C(8'')-C(9'')-C(10'') & 132 \\ C(1')-O(1')-C(1') & 111-6 & (9) & O(4')-C(12')-O(5') & 125 \\ C(4')-O(2')-C(7') & 120 & (1) & O(4')-C(12')-C(13') & 1125 \\ \end{array}$	C(2')-C(3')-C(4')	110 (1)	O(3")-C(7")-C(8")	124 (2)
$\begin{array}{llll} C(4')-C(3')-O(2') & 108 (1) & C(7')-C(8')-C(11') & 118 \\ C(3')-C(4')-C(5') & 112 (1) & C(9')=C(8')-C(11') & 130 (1) \\ C(3')-C(4')-O(2') & 108 (1) & C(7'')-C(8'')=C(9'') & 129 (1) \\ O(2')-C(4')-C(5') & 104 (1) & C(7'')-C(8'')-C(11'') & 106 (1) \\ C(4')-C(5')-C(6') & 113 (1) & C(9'')=C(8'')-C(11'') & 125 (1) \\ C(4')-C(5')-O(1') & 108 (1) & C(8')=C(9')-C(10'') & 134 (1) \\ C(8')-C(5')-O(1') & 108 (1) & C(8'')=C(9'')-C(10'') & 132 (1) \\ C(1')-O(1')-C(1') & 111-6 (9) & O(4')-C(12')=O(5') & 125 (1) \\ C(4')-O(2')-C(7') & 120 (1) & O(4')-C(12')-C(13') & 112 (1) \\ \end{array}$	C(2')-C(3')-O(2'')		C(7')-C(8')=C(9')	112 (2)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$				118 (2)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(3')-C(4')-C(5')		C(9')=C(8')-C(11')	130 (2)
$\begin{array}{llll} O(2')-C(4')-C(5') & 104 & (1) & C(7'')-C(8'')-C(11'') & 106 \\ C(4')-C(5')-C(6') & 113 & (1) & C(9'')-C(8'')-C(11'') & 125 \\ C(4')-C(5')-O(1') & 108 & (1) & C(8'')-C(9')-C(10'') & 134 \\ C(8')-C(5')-O(1') & 108 & (1) & C(8'')-C(9'')-C(10'') & 132 \\ C(1')-O(1')-C(1') & 111-6 & (9) & O(4')-C(12')-O(5') & 125 \\ C(4')-O(2')-C(7') & 120 & (1) & O(4')-C(12')-C(13') & 1121 \\ \end{array}$			C(7'')-C(8'')=C(9'')	129 (2)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$			C(7")-C(8")-C(11")	106 (2)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$				125 (2)
C(8')-C(5')-O(1') 108 (1) $C(8'')-C(9'')-C(10'')$ 132 (1) $C(1')-O(1')-C(1')$ 111-6 (9) $O(4')-C(12')=O(5')$ 125 (1) $C(4')-O(2')-C(7')$ 120 (1) $O(4')-C(12')-C(13')$ 112 (1)				
C(1')-O(1')-C(1') 111-6 (9) $O(4')-C(12')=O(5')$ 125 (C(4')-O(2')-C(7') 120 (1) $O(4')-C(12')-C(13')$ 112 (1)	C(4')-C(5')-O(1')			
C(4')-O(2')-C(7') 120 (1) $O(4')-C(12')-C(13')$ 112 (C(8'')=C(9'')-C(10'')	132 (2)
C(4')-O(2')-C(7') 120 (1) $O(4')-C(12')-C(13')$ 112 (O(4')-C(12')=O(5')	125 (1)
	C(4')-O(2')-C(7')	120 (1)	O(4')C(12')C(13')	112(1)
C(3')-O(2'')-C(7'') 114 (1) $O(5')=C(12')-C(13')$ 123 (C(3')-O(2")-C(7")	114 (1)	O(5')=C(12')-C(13')	123 (2)

In the triterpene skeleton, several C-C bonds [C(1)-C(10); C(5)-C(10); C(8)-C(14); C(9)-C(10);C(17)-C(22) deviate from the mean value of 1.54 Å. it seems significant that all these long C-C bonds are attached to at least one of the fully substituted carbon atoms [C(8), C(10), C(14) and C(17)]. Several C-C-C angles also deviate from the accepted value of 109.3°. The degrees of substitution of the carbon

atoms and long-range steric strain in the structure (s below) seem to be responsible for these deviations.

The C(12)–C(13) bond [1.37 (2) Å] is shorter that the remainder of the ring-system bonds and com sponds to the expected location of the double bond oleanene triterpenes.

In Table 4, the conformation of the ring systemi analyzed in terms of the least-squares planes an interplanar angles. The equations of the least-square planes were calculated by the method of Schomaka (6)-(Waser, Marsh & Bergman (1959). The torsional angle (5)-(in the ring system are given in Table 5(i). From Table 5(i). 4 and 5, it is clear that the rings A, B, D and E take $\mathbb{C}(\eta)$ -(

Table 4. Least-squares planes

(a) Equations of the planes
Plane A C(1), C(2), C(3), C(4), C(5), C(10)
-0.8682x - 0.4203y - 0.2637z + 9.6570 = 0
Plane B $C(5)$, $C(6)$, $C(7)$, $C(8)$, $C(9)$, $C(10)$
-0.8343x - 0.5346y + 0.1344z + 9.2884 = 0
Plane C C(8), C(9), C(11), C(12), C(13), C(14)
-0.7409x - 0.6612y + 0.1178z + 7.8474 = 0
Plane D C(13), C(14), C(15), C(16), C(17), C(18)
-0.8455x - 0.5273y - 0.0838z + 10.3836 = 0
Plane $E \in C(17)$, $C(18)$, $C(19)$, $C(20)$, $C(21)$, $C(22)$
-0.8856x + 0.1556y + 0.4376z + 9.1570 = 0
Plane $F \in C(21), O(11), C(1')$
0.0951x - 0.9953y - 0.0203z - 2.6655 = 0
Plane $G \in C(1'), C(2'), C(3'), C(4'), C(5'), O(1')$
-0.4992x - 0.6567y + 0.5653z + 1.0736 = 0

(b) Deviations (A) from the least-squares planes

Di	,	Diana I	9	Dlama C	, P	dista
Plane A		Plane E		Plane C		<u> </u>
C(1)	-0.24(1)	C(5)	-0.50(1)	C(8)	0.38(1)	
C(2)	0.25 (2)	C(6)	0.15(1)	C(9)	-0.29 (1)	
C(3)	-0·24 (2)	C(7)	−0 ·15 (1)	C(11)	0.04(2)	here
C(4)	0.22(1)	C(8)	0.39(1)	C(12)	0.09(1)	
C(5)	-0.23(1)	C(9)	-0.08(1)	C(13)	-0.004 (1)	Т
C(10)	0.23(1)	C(10)	0.19(1)	C(14)	-0·23 (I)	
O(1)	0.26(1)	C(25)	1.66 (2)	C(26)	1.88(1)	
C(23)	-0.56(2)	C(26)	1-89 (1)	C(27)	-1·77(I)	at (
C(24)	1·69 (1)	` ,	• •	• •		inter
C(25)	1·75 (2)					shov
- (/	(-/					
					-3	Time-
Plane L)	Plane E	3	Plane G	* / (I	ring
Plane <i>L</i> C(13)					_0·26 (I)	towa
C(13)	0.11(1)	C(17)	-0.13 (1)	C(1')		towa
C(13) C(14)	0·11 (1) -0·12 (1)	C(17) C(18)	-0·13 (1) 0·18 (1)	C(1') C(2')	-0·26 (I) 0·22 (I)	towa C(2)
C(13) C(14) C(15)	0·11 (1) -0·12 (1) 0·17 (1)	C(17) C(18) C(19)	-0·13 (1) 0·18 (1) -0·27 (1)	C(1') C(2') C(3')	_0.26 (i)	towa C(2) rings
C(13) C(14) C(15) C(16)	0·11 (1) -0·12 (1) 0·17 (1) -0·23 (1)	C(17) C(18) C(19) C(20)	-0·13 (1) 0·18 (1) -0·27 (1) 0·30 (1)	C(1') C(2') C(3') C(4')	-0.26 (I) 0.22 (I) -0.21 (I) 0.21 (I)	towa C(2) rings The
C(13) C(14) C(15) C(16) C(17)	0·11 (1) -0·12 (1) 0·17 (1) -0·23 (1) 0·21 (1)	C(17) C(18) C(19) C(20) C(21)	-0·13 (1) 0·18 (1) -0·27 (1) 0·30 (1) -0·26 (1)	C(1') C(2') C(3') C(4') C(5')	-0.26 (I) 0.22 (I) -0.21 (I) 0.21 (I) -0.24 (I)	towarings C(2) The
C(13) C(14) C(15) C(16) C(17) C(18)	0·11 (1) -0·12 (1) 0·17 (1) -0·23 (1) 0·21 (1) -0·16 (1)	C(17) C(18) C(19) C(20) C(21) C(22)	-0·13 (1) 0·18 (1) -0·27 (1) 0·30 (1) -0·26 (1) 0·19 (1)	C(1') C(2') C(3') C(4') C(5') O(1')	-0.26 (l) 0.22 (l) -0.21 (l) 0.21 (l) -0.24 (l) 0.28 (l)	lowa C(2) Tings The Ting
C(13) C(14) C(15) C(16) C(17) C(18) C(27)	0·11 (1) -0·12 (1) 0·17 (1) -0·23 (1) 0·21 (1) -0·16 (1) -1·63 (1)	C(17) C(18) C(19) C(20) C(21) C(22) C(28)	-0·13 (1) 0·18 (1) -0·27 (1) 0·30 (1) -0·26 (1) 0·19 (1) 0·83 (1)	C(1') C(2') C(3') C(4') C(5') O(1') O(11)	-0.26 (l) 0.22 (l) -0.21 (l) 0.21 (l) -0.24 (l) 0.28 (l) 0.15 (l)	toward C(2) Tings The ring axial
C(13) C(14) C(15) C(16) C(17) C(18) C(27) O(5)	0·11 (1) -0·12 (1) 0·17 (1) -0·23 (1) 0·21 (1) -0·16 (1) -1·63 (1) -1·71 (1)	C(17) C(18) C(19) C(20) C(21) C(22) C(28) C(29)	-0·13 (1) 0·18 (1) -0·27 (1) 0·30 (1) -0·26 (1) 0·19 (1) 0·83 (1) -0·29 (1)	C(1') C(2') C(3') C(4') C(5') O(1') O(11) O(4')	-0.26 (J) 0.22 (J) -0.21 (J) 0.21 (J) -0.24 (J) 0.28 (J) 0.15 (J) -0.40 (J)	toward C(2) The ring axial C(2)
C(13) C(14) C(15) C(16) C(17) C(18) C(27)	0·11 (1) -0·12 (1) 0·17 (1) -0·23 (1) 0·21 (1) -0·16 (1) -1·63 (1)	C(17) C(18) C(19) C(20) C(21) C(22) C(28) C(29) C(30)	-0·13 (1) 0·18 (1) -0·27 (1) 0·30 (1) -0·26 (1) 0·19 (1) 0·83 (1) -0·29 (1) 1·81 (1)	C(1') C(2') C(3') C(4') C(5') O(1') O(11) O(4') O(2")	-0.26 (I) 0.22 (I) -0.21 (I) 0.21 (I) -0.24 (I) 0.28 (I) 0.15 (I) -0.40 (I) 0.54 (I)	C(2) The ring axial C(2)
C(13) C(14) C(15) C(16) C(17) C(18) C(27) O(5)	0·11 (1) -0·12 (1) 0·17 (1) -0·23 (1) 0·21 (1) -0·16 (1) -1·63 (1) -1·71 (1)	C(17) C(18) C(19) C(20) C(21) C(22) C(28) C(29)	-0·13 (1) 0·18 (1) -0·27 (1) 0·30 (1) -0·26 (1) 0·19 (1) 0·83 (1) -0·29 (1)	C(1') C(2') C(3') C(4') C(5') O(1') O(11) O(4')	-0.26 (J) 0.22 (J) -0.21 (J) 0.21 (J) -0.24 (J) 0.28 (J) 0.15 (J) -0.40 (J)	C(2) The Ting The ming axia C(2)

(c) Interplanar angles (°)

A/B	156.0 (4)	D/E	129-1 (3)
B/C	170.9 (4)	E/F	75.6 (2)
C/D	164.9 (4)	F/G	126-5 (3)

chair **co**nfc **C**(12 rings E rin distor distor action C(10 Other

the re betwe

value

C(9)-(**E**(11)–

distar ml xi: 'n rŧ

2

Table 5. Torsional angles (°)

	G(0) G(1) G(13)	27.0(1)	(ii) Chappyrapocul ring	
		, ,		50 6 (2)
55.6 (2)	C(9)-C(11)-C(12)-C(13)	6-5 (1)		58.6 (2)
58-1 (2)	C(11)-C(12)-C(13)-C(14)	2.0(1)		-67.1(3)
54.8 (2)	C(12)-C(13)-C(14)-C(15)	-150·0 (3) '		-54.2 (2)
• •	C(18)-C(13)-C(14)-C(15)	32.3 (2)	C(2')-C(3')-C(4')-C(5')	-53.0(2)
• •	C(14)-C(13)-C(18)-C(17)	-35.8(2)	C(3')-C(4')-C(5')-O(1')	-56.9(2)
-165.5(4)	C(13)-C(14)-C(15)-C(16)	37.4 (2)	C(4')-C(5')-O(1')-C(1')	63.8 (3)
171.9 (3)	C(14)-C(15)-C(16)-C(17)	48.5 (3)		
61.1 (2)	C(15)-C(16)-C(17)-C(18)	50.4 (3)	(iii) Angelyl groups	
60-3 (2)	C(16)-C(17)-C(18)-C(13)	42.9 (3)		113.4 (3)
50.4 (2)	C(16)-C(17)-C(18)-C(19)	83.5 (3)		-11.60 (2)
50-5 (2)	C(22)-C(17)-C(18)-C(13)	$-169 \cdot 1 (3)$		-19.54(2)
179·7 (4)	C(22)-C(17)-C(18)-C(19)	42.6 (3)		171-14 (3)
169-1 (3)	C(18)-C(17)-C(22)-C(21)	43.1 (2)		106.8 (4)
-61.7(2)	C(17)-C(18)-C(19)-C(20)	56.7 (3)		144-3 (4)
56.3 (1)	C(18)-C(19)-C(20)-C(21)	−62·2 (3)		149-1 (3)
61.5 (2)	C(19)-C(20)-C(21)-C(22)	59-6 (3)	C(11')C(8')C(9')C(10')	174-6 (4)
	58·1 (2) 54·8 (2) -53·6 (2) 64·9 (2) -165·5 (4) 171·9 (3) 61·1 (2) 50·3 (2) 50·5 (2) 179·7 (4) 169·1 (3) -61·7 (2) 56·3 (1)	58·1 (2) C(11)—C(12)—C(13)—C(14) 54·8 (2) C(12)—C(13)—C(14)—C(15) -53·6 (2) C(18)—C(13)—C(14)—C(15) 64·9 (2) C(14)—C(13)—C(18)—C(17) -165·5 (4) C(13)—C(14)—C(15)—C(16) 171·9 (3) C(14)—C(15)—C(16)—C(17) 61·1 (2) C(15)—C(16)—C(17)—C(18) 60·3 (2) C(16)—C(17)—C(18)—C(13) 50·4 (2) C(16)—C(17)—C(18)—C(19) 50·5 (2) C(22)—C(17)—C(18)—C(19) 179·7 (4) C(22)—C(17)—C(18)—C(19) 169·1 (3) C(18)—C(17)—C(22)—C(21) -61·7 (2) C(17)—C(18)—C(19)—C(20) 56·3 (1) C(18)—C(19)—C(20)—C(21)	55.6 (2) C(9)—C(11)—C(12)—C(13) 6.5 (1) 58.1 (2) C(11)—C(12)—C(13)—C(14) 2.0 (1) 54.8 (2) C(12)—C(13)—C(14)—C(15) —150.0 (3) —53.6 (2) C(18)—C(13)—C(14)—C(15) 32.3 (2) 64.9 (2) C(14)—C(13)—C(18)—C(17) —35.8 (2) —165.5 (4) C(13)—C(14)—C(15)—C(16) 37.4 (2) 171.9 (3) C(14)—C(15)—C(16)—C(17) 48.5 (3) 61.1 (2) C(15)—C(16)—C(17)—C(18) 50.4 (3) 60.3 (2) C(16)—C(17)—C(18)—C(13) 42.9 (3) 50.4 (2) C(16)—C(17)—C(18)—C(13) 42.9 (3) 50.5 (2) C(22)—C(17)—C(18)—C(13) —169.1 (3) 179.7 (4) C(22)—C(17)—C(18)—C(19) 42.6 (3) 169.1 (3) C(18)—C(17)—C(22)—C(21) 43.1 (2) —61.7 (2) C(17)—C(18)—C(19)—C(20) 56.7 (3) 56.3 (1) C(18)—C(19)—C(20)—C(21) —62.2 (3)	$\begin{array}{cccccccccccccccccccccccccccccccccccc$

conformation while the C ring adopts a half-chair formation as a result of the double bond between (12) and C(13). The junctions of the A/B and B/Care in the trans form while that between the D and mgs is cis. The rings B, D and E are significantly lotted as is the whole molecular skeleton. These fortions seem to be caused mainly by the intertions between the bulky axial groups (at C(4), C(8), (0) on one hand and at C(14) and C(16) on the which push each other away in order to release repulsive forces. Actually, the non-bonded distances ween 1,3 diaxial groups are larger than the expected in non-distorted rings. We have reported these Inces in Table 6. In a regular chair-form sixmbered ring, the non-bonded distances between 1,3 rial groups would be 2.52 Å. These distances are increased to, for example, 3.27 (2) Å [C(24) C(25)].

itions.
horter that and correble bond in

g system in planes and cast-square Schomaker ional angle rom Table and E take

10

0

0 = 0

0

0

0

C

0·38 (1) -0·29 (1) 0·04 (2)

0·09 (1) -0.004 (1)

_0·23 (I)

1.88(1)

-0·26 (I)

0·22 (I) -0·21 (I)

-0.52 (1)

0.28 (2)

29-1 (3)

15-6 (2)

The distortion of ring C from a regular half-chair formation may be due to the degree of substitution C(8) and C(14). Also resulting from 1,3 diaxial faction is the distortion of the whole molecule. As no by the angles between the mean planes of the A, B, C and D, the triterpene skeleton is convex for the axial groups C(24), C(25), C(26) and C(25). On the other hand there is twisting between E and D about the long direction of the molecule. The same of the steric effect of the three bulky for acetate groups situated at C(16), C(17) and A similar conformation of the triterpenic signals has been previously observed in gymnematory Hoge & Nordman (1974).

In glucopyranosyl ring has an almost regular chair formation with all the substituent groups in the local positions. The bond distances agree with the reported for β -D-glucose (Chu & Jeffrey, 1968)

Table 6. Some non-bonded distances (Å) between 1,3 diaxial groups

C(24)C(25)	3.27 (2)
C(25)C(26)	3.20(2)
C(27)O(5)	3.06(2)
O(5)O(9)	2.97(1)

Table 7. Shortest intermolecular distances (Å) with standard deviations in parentheses

Key to symmetry operations relating designated atoms to reference atoms at (x,y,z):

y, ž	(iv) $\ddot{x} + 1, \frac{1}{2} + y, \ddot{z} + 1$ (v) $x, y - 1, z$ (vi) $\ddot{x} + 2, \frac{1}{2} + y, \ddot{z} + 2$.		
3.56 (3)	$C(30)\cdots O(2^{iii})$	3.63 (2)	
3.69 (2)	$C(32)\cdots O(4^{iv})$	3.10 (3)	
3.60(2)	$C(34)\cdots O(2^{\nu})$	3.30 (3)	
3-35 (2)	$C(36)\cdots C(40^{11})$	3.57 (3)	
3.60(2)	$C(38)\cdots O(4^{l})$	3.58 (3)	
3.56(2)	$O(1)\cdots O(8^l)$	3.36 (2)	
	$O(6)\cdots C(3^{\prime li})$	3.46 (2)	
	$O(6)\cdots C(5'^{li})$	3.68 (2)	
3.55 (2)	$C(13')\cdots O(3'^{vl})$	3.54 (3)	
	3.56 (3) 3.69 (2) 3.60 (2) 3.35 (2) 3.56 (2) 3.56 (2) 3.52 (3) 3.61 (3)	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	

with one exception: the C(3')-C(4') bond is significantly shorter in comparison with the accepted value of 1.54 Å. Moreover, the two angles C(2')-C(3')-O(2'') and C(5')-C(4')-O(2') differ from the tetrahedral angle of 109.5° . Steric strain resulting from the two long angelyl side chains situated at C(3') and C(4') could be responsible for these unusual values. Indeed, these angelyl groups, in order to overcome any overlapping, are rotated with respect to each other by

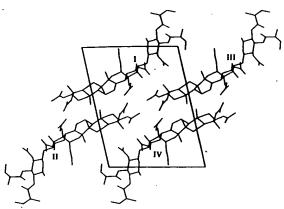


Fig. 3. Projection of the acetylated napoleogenin structure along the b axis. The positions of the molecules are; (I) at x, y, z; (II) at $\bar{x} + 1$, $\frac{1}{2} + y$, z; (III) at x, y, z + 1; (IV) at x + 1, $\frac{1}{2} + y$, $\bar{z} + 1$ with x, y and z coordinates given in Table 1.

about 180° about the C(7')—C(8') bond [the corresponding torsional angles are reported in Table 5(iii)].

One should also notice that in both angelyl and acetyl groups, the accuracy of bond lengths and valency angles is highly restricted by a large thermal motion of the end-chain atoms.

A packing diagram of the crystal structure viewed along the b axis is shown in Fig. 3. The intermolecular distances less than 3.7 Å are reported in Table 7. There is no abnormal feature in the packing, which appears to

be dictated mainly by close van der Waals containeida between the molecules.

The authors wish to thank Professors J. Tousse amin and A. Van de Vorst for their interest in this work independent Mr. Vermeire for technical assistance. Crystals acetylated napoleogenin were provided by the H. Laboratoire de Chimie Organique of Professor R. H. Essum

References

CHU, S. S. C. & Jeffrey, G. A. (1968). Acta Cryst. В 830-838.

CRUICKSHANK, D. W. J. (1961). Computing Methods the Phase Problem in X-ray Crystal Analysis, edited by Pepinsky, J. M. Robertson & J. C. Speakman. Oxfo Pergamon Press.

Crystallographic Data Files (1979). PLUTO. Crystal graphic Data Centre, Univ. Chemical Laborator Cambridge, England.

Hoge, R. & Nordman, C. E. (1974). Acta Cryst. Bl 1435-1440.

MAIN, P., LESSINGER, L., WOOLFSON, M. M., GERMAIN, Spe & DECLERCQ, J. P. (1977). MULTAN 77. A System 0.40: Computer Programs for the Automatic Solution of Cry Structures from X-ray Diffraction Data. Univ. of Yamultip England.

PREWITT, C. T. (1967). SFLS. State Univ. of New York Stony Brook, Long Island, New York 11794, USA.

SCHOMAKER, V., WASER, J., MARSH, R. E. & BERGMAN, (1959). Acta Cryst. 12, 600-604.

Acta Cryst. (1980). B36, 1598-1601

The Structure of 2-Amino-5-nitrophenol: a Comparison with 2-Aminophenol

By Masao Haisa, Setsuo Kashino and Takayuki Kawashima

Department of Chemistry, Faculty of Science, Okayama University, Tsushima, Okayama 700, Japan

(Received 23 October 1979; accepted 11 February 1980)

Abstract

The crystal structure of 2-amino-5-nitrophenol has been determined from visually estimated Cu $K\alpha$ data, and refined to an R value of 0.072 for 1138 non-zero reflexions. The crystals are monoclinic, $P2_1/c$, with a=8.49 (1), b=10.34 (1), c=8.05 (1) Å, $\beta=113\cdot1$ (1)° and Z=4. The molecules, related by a glide plane, are held together by $O-H\cdots NH_2$ hydrogen bonds $[O\cdots N 2.801$ (3), $H\cdots N 1.89$ (4) Å, $O-H\cdots N 174$ (3)°] to form a ribbon along c. The ribbons are held together by dipole—dipole interactions

0567-7408/80/071598-04\$01.00

between the molecules related by 1, as found of monly in mononitrophenols. The morphotropism mononitrophenols and aminophenols is discussed.

Introduction

As part of a programme of studies on the crystal molecular structures of phenols (Kagawa, Kashino & Haisa, 1976) and the systemization organic crystals (Haisa, 1978), the structure of amino-5-nitrophenol has been determined in order

© 1980 International Union of Crystallography

frown $\{100\}$ $\{C_6H_6\}$ sition $\{C_6H_6\}$ $\{C_6H_6\}$

Erysta

Cu Ka Spe 0.40 photol multip graphs from / (78% observ visuall and fo

Pbca, V = 1 The ro Pant (

The st the H map w blocking sc 0 < 1/2 extinct

> = 0 on-z₍ sted i

Lis ave be uppler blaine rystal

This Page is Inserted by IFW Indexing and Scanning Operations and is not part of the Official Record

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

BLACK BORDERS

☑ BLACK BORDERS
MIMAGE CUT OFF AT TOP, BOTTOM OR SIDES
FADED TEXT OR DRAWING
☐ BLURRED OR ILLEGIBLE TEXT OR DRAWING
☐ SKEWED/SLANTED IMAGES
COLOR OR BLACK AND WHITE PHOTOGRAPHS
☐ GRAY SCALE DOCUMENTS
☐ LINES OR MARKS ON ORIGINAL DOCUMENT
☐ REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY
П отнер.

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.